A. PROJECT MANAGEMENT

A1. Title and Approval Sheet

U.S. Environmental Protection Agency
Office of Research and Development
Center for Environmental Measurement and Modeling
Watershed & Ecosystem Characterization Division
Watershed Management Branch

Quality Assurance Project Plan

Title: QAPP FOR SURVEY OF RESERVOIR GREENHOUSE GAS EMISSIONS (SuRGE)

QA Category: ☐ A ☒ B

ORD National Program Project/Task ID: AE.2.6 Methods for estimating methane emissions from surface water reservoirs for the U.S. GHG Inventory Report

QAPP was Developed: ☒ Intramurally ☐ Extramurally:

QAPP Accessibility: QAPPs will be made internally accessible via the [ORD QAPPs intranet site] upon final approval *unless the following statement is selected*.

☐ I do NOT want this QAPP internally shared and accessible on the ORD intranet site.

Project Type(s) (check all that apply):

☒ Environmental Measurements ☐ Environmental Technology ☐ Decision Support Tool ☐ Existing Data ☐ Informatics ☐Geospatial ☐ Method Development ☐ Model Application ☐ Model Development ☐ Software and Data Management ☐Remote Sensing ☐ Technical Assessment ☐ Other

Approvals

Prepared by: Dr. Jake J. Beaulieu	JAKE BEAULIEU Digitally signed by JAKE BEAULIEU Date: 2022.05.18 09:00:35 -04'00'	
	Signature	*Date*
Branch Chief: Dr. Michael Elovitz	MICHAEL ELOVITZ Digitally signed by MICHAEL ELOVITZ Date: 2022.05.18 09:05:15 -04'00'	
	Signature	*Date*
QA Manager: Margie Vazquez	MARGARITA VAZQUEZ Digitally signed by MARGARITA VAZQUEZ Date: 2022.05.18 09:28:30 -04'00'	
	Signature	*Date*

A2. Table of Contents

Figures

Tables

QAPP Revision History			
QAPP ID Number	**Prepared By**	**Date of Revision**	**Description of Change**
J-WECD-0032592-QP-1-0	Jake Beaulieu	03/31/2020	Original QAPP.
J-WECD-0032592-QP-1-1	Jake Beaulieu	06/15/2020	Revised to include additional details on GPS site tracking, addition of Sections F14. PICARRO GREENHOUSE GAS ANALYZER QA/QC SHEET and F15. Bad Elf GPS USER GUIDE, more details regarding equipment and supplies (Section F8. SuRGE EQUIPMENT AND SUPPLIES), and other data collection details.
J-WECD-0032592-QP-1-2	Jake Beaulieu	09/08/2020	G. Addendum was added to discuss the effect of COVID 19 shutdowns and safety measures on sampling and analyte hold times. Other minor modifications made, such as to file naming conventions, to base kit list, to Appendix F9. SuRGE WATER CHEMISTRY TRACKING SHEET, as well as other minor edits.
J-WECD-0032592-QP-1-3	Jake Beaulieu	05/21/2021	• Added algal taxonomy and physiology. • Added DOC and anions. • Specify that sonde records bulk chlorophyll, not just the chlorophyll a pigment. • Added alternate shipping contacts. • Clarified data management and file naming conventions • Minor data sheet updates. • Added reference to training videos
J-WECD-0032592-QP-1-4	Jake Beaulieu	06/04/2021	• Updated chemistry CoC form (section F9. SuRGE WATER CHEMISTRY TRACKING SHEET) to include DOC and total anions. • Fixed a mistake in Figure 4 • Added analyzer time zone to F3. SuRGE GREENHOUSE GAS

			ANALYZER AND GPS DATA SHEET.
J-WECD-0032592-QP-1-5	Jake Beaulieu	11/30/21	• Specify not passive trap deployment at depths > 80'. • Added cell pressure field to F13. LOS GATOS GREENHOUSE GAS ANALYZER QA/QC SHEET. • Changing total anions to dissolved anions. • Added option for Geopump cartridge filter • Specify ADA analyzes filtered anions in-house • Additional guidance for Sample Tracking Forms and Chain of Custody Forms • Geoplatform log-in procedure for non EPA collaborators • Added qa.qc status field to F1.
J-WECD-0032592-QP-1-6	Jake Beaulieu	April 2022	• Added field to sample tracking sheets for technician to note sample condition when received from field, but before shipped to analytical laboratory. • Guidance on TTEB and Lachat lab Chain of Custody forms. • Added picture of properly fixed taxonomy sample. • Specified filter membrane in table 5. • Added SUVA • Added 'Trip_ID`, `QA.QC lake` and 'reservoir outflow' fields to F1. • Added sonde based phycocyanin • Added SOP references for EXO sonde. • Updated Los Gatos cell pressure specifications
J-WECD-0032592-QP-1-7	Jake Beaulieu	May 2022	• Changed primary contact for NAR lab to Stephen Shivers. • Updated Explorer login procedure.

A3. Distribution List

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A4. Project Organization

Project Technical Lead: Dr. Jake Beaulieu (ORD/CEMM/WECD) will be the technical lead person for the project. His responsibilities include:

- Authoring and maintaining QAPP and relevant SOPs
- Generating overall survey design
- Generating survey designs for each waterbody.
- Serving as Contracting Officer Representative on Work Assignment / Task Order for Cincinnati on-site contract support.
- Coordinating project analytics (e.g., water chemistry) in AWBERC laboratories.
- Coordinating equipment needs among field crews.
- Ensuring that Field Crew Leads are exercising sufficient oversight over field activities.
- Data analysis and reporting

Field Crew Technical Lead: Field crews will be deployed from at least six geographic locations.

- Each field crew will have a technical lead whose responsibilities include:
    - Ensuring site access is coordinated with relevant governing authorities (e.g., private property owners, park managers) and that necessary permits/exemptions are obtained.
    - Coordinating sampling schedule with Project Technical Lead.
    - Ensuring that field personnel adhere to procedures detailed in QAPP and SOPs
    - Oversee data management for sites sampled by their field crew.
- Field Crew Technical Leads are:
    - Cincinnati: Scott Jacobs (ORD/CESER/WID)
    - Narragansett: Jeff Hollister (ORD/CEMM/ACESD)
    - RTP: John Walker (ORD/CEMM/AESMD)
    - ADA: Ken Forshay (ORD/CESER/GCRD)
    - USGS: Bridget Deemer
    - R10: Lil Herger (USEPA/R10/LSASD)

Research Support Staff: Each Field Crew Technical Lead will be supported by 'Research Support Staff' who will be responsible for executing the procedures described in the QAPP and SOPs. Research Support Staff may vary from lake to lake, or year to year, but they must read and understand the QAPP and SOPs prior to contributing to the project. Research Support Staff should work in close coordination with Field Crew Technical Lead to ensure resources are coordinated appropriately and procedures are implemented in accordance with QA/QC documentation.

Gas Lab Technical Lead: Karen White (ORD/CEMM/WECD) will be the technical lead for the analysis of gas samples generated by the field crews. Responsibilities include:

- Evacuating and labeling gas vials for field sampling.
- Analyzing gas samples.
- Managing data from gas analyses.
- Submitting procurement requests for GC consumables.

Water Chemistry Technical Leads: Each laboratory will have a water chemistry technical lead responsible for 1) ensuring that samples are received, stored, and analyzed according the relevant SOPs and QAPP, 2) ensuring that analytical data meet the relevant QA/QC criteria, or are flagged to indicate non-compliance, and 3) delivering the final data to the specified electronic repository (B.3.5 Electronic records).

- Cincinnati labs: Jake Beaulieu (ORD/CEMM/WECD)
    - Water chemistry through onsite Pegasus contract, SEEs, and Fed staff: dissolved anions, dissolved nutrients, total nutrients, DOC, TOC, and dissolved metals
- Ada labs: Ken Forshay (ORD/ CESER/GCRD)
    - Water chemistry for samples taken by Ada field crew: dissolved nutrients, total nutrients, DOC, TOC, and dissolved anions. Metals will be shipped to Cincinnati.
- Narragansett labs: Stephen Shivers (ORD/ CEMM/ACESD)
    - Algal indicators: chlorophyll a, phycocyanin, and microcystin
- Gulf Breeze labs: Avery Tatters (ORD/CEMM/GEMMD)
    - Algal taxonomy and physiology

Quality Assurance Manager: Margie Vazquez (ORD/CEMM/WECD) will provide independent quality assurance oversight to ensure that planning and plan implementation are in accordance with approved QAPP. The Quality Assurance Manager will provide technical direction from QA/QC perspective and will enter QAPP and all related products into the ORD QA database QA Track.

A5. Problem Definition and Background

Humans have built reservoirs for hydroelectric power generation, flood control, drinking water sources, and other uses. These man-made systems have provided society with important services, but these have come at the cost of enhanced greenhouse gas (GHG) emissions resulting from the biological production of carbon dioxide (CO_2), methane (CH_4), and nitrous oxide (N_2O) in reservoir waters and sediments. The Intergovernmental Panel on Climate Change (IPCC) recently approved a methodology for including this emission source in national greenhouse gas inventories and the EPA's Office of Air and Radiation has requested ORD support in its implementation.

To reduce uncertainty in the inventory estimate, OAR has requested that ORD develop empirically based emission factors for GHG emissions from U.S. reservoirs. Currently, there are insufficient data to develop emission factors for the U.S., therefore ORD and partners will conduct a national-scale survey of U.S. reservoirs during the summers of FY20 – FY23. This four-year field campaign will generate the data needed to define empirically based emission factors allowing for an accurate and well constrained emission estimate for inclusion in the Inventory.

The primary work-product generated from this effort will be the data-set from the national-scale survey. The survey will not conclude until after the end of the current StRAP cycle

(September 2023), however, and analysis, presentation, and publication of the data will be conducted under the FY23-26 StRAP (Table 1). In the interim, ORD will develop a baseline estimate of methane emissions from U.S reservoirs using default IPCC emission factors. This baseline estimate will be revised during the FY19-22 StRAP as field data from the survey become available.

A6. Project/Task Description

	2020		2021	2022	2023	2023	2024
	Spring	Summer	Summer	Summer	Summer	Fall/Winter	Winter/Spring
Activity							
Method development/survey design							
Sample collection							
Data analysis							
Writing							

Table 1. Project schedule

A7. Quality Objectives and Criteria for Measurement Data

The objective of the study is to estimate GHG emissions from U.S. reservoirs with a 95% confidence interval of +/- 25% of the estimated value. A power analysis using previously collected data suggested that approximately 100 measurement sites would be required to meet this criterion. The survey design includes 108 sites evenly allocated among the nine major U.S. ecoregions.

A8. Special Training/Certifications

COVID travel restrictions have made it difficult to organize a centralized training session for field methodologies. The RTP and R10 field crews have prior experience with the sampling methods and can sample without additional training. Dr. Beaulieu provided on-site training for the CIN field crew in June 2020, prior to the 2020 field work. ADA, USGS, and NAR field crews require training. The USGS will attend a training in Cincinnati in May 2021. ADA field crew will self-train utilizing the detailed guidance found in the QAPP and demonstration videos. The Project Technical Lead, CIN Field Crew Technical Lead, or CIN Research Support Staff will travel to NAR to provide on-site training during the summer of 2021. After COVID restrictions have eased, the Project Technical Lead, or an experienced Field Crew Technical Lead, will conduct site visits to provide additional on-site training and oversight.

Boat operators may be required to receive training from the relevant state agency (e.g., Ohio Department of Natural Resources). The Field Crew Technical Leads should verify the state requirements for their sampling area. Electronic copies of the certificates will be stored at the SuRGE Sharepoint documents library: "Environmental Protection Agency (EPA)\SuRGE Survey of Reservoir Greenhouse gas Emissions - Documents\projectDocuments\training certificates"

A9. Documents and Records

Paper records will be generated per the data sheets in section F. Appendix. Paper and electronic records will be managed as specified in B.3.4 Paper records and B.3.5 Electronic records.

B. DATA GENERATION AND ACQUISITION

B1. Experimental Design

B1.1 Survey design

The objective of the research is to estimate the magnitude of GHG emissions from U.S. reservoirs. Accomplishing this via classical survey design principles requires 1) a definitive list of all U.S. reservoirs (the 'sample frame'), and 2) a probabilistic subset of the sample frame for sampling. No definitive list of U.S. reservoirs exists, however, so the sample frame for SuRGE is defined as the 522 reservoirs sampled in EPA's 2017 National Lakes Assessment (NLA). The NLA is a probabilistic survey of all U.S. waterbodies > 1 Ha included in the USGS National Hydrography Dataset (NHDPlusV2). The subset of NLA sites that are confirmed to be reservoirs by the field crews therefore represent a probabilistic subset of all U.S. reservoirs. This property of the NLA sampled reservoirs will allow us to estimate GHG emissions for all U.S. reservoirs by making detailed measurements at a subset of the NLA reservoirs.

Upscaling measurements to the nation can also be accomplished via statistical modeling based on environmental drivers that control emission rates. To ensure variation in potential environmental drivers across the reservoirs, and to ensure national-scale coverage, the survey design entails stratification by ecoregion, chlorophyll a, and depth. Twelve sites are distributed within each of the 9 major U.S. ecoregions, for a total of 108 sample sites. Reservoirs are further classified as high or low chlorophyll a (> or < 7 ug/L, respectively) and deep or shallow (> or < 6m, respectively), creating four unique combinations of depth and chlorophyll a. Within each ecoregion, three sites are apportioned to each of these four combinations. The survey design also includes 'oversample' sites which can be used to replace main sites that are inaccessible due to landowner denial, lack of physical access, or other reasons.

The 108 reservoirs (Figure 1) will be sampled one-time between June 1 and Sept. 15 of 2020, 2021, 2022, or 2023. Within each reservoir, sampling will consist of measurements of diffusive and ebullitive emissions at ≥15 sites. Water chemistry will be sampled at the site where we anticipate the greatest depth, defined as the 'Index Site'. Larger, or highly dendritic reservoirs, may be broken into several sections. This is done to ensure that Oversample sites, when needed, are always within a reasonable distance of the target site that is being replaced. The location of the sampling sites will be defined using a Generalized Random Tessellation Stratified (GRTS) survey design.

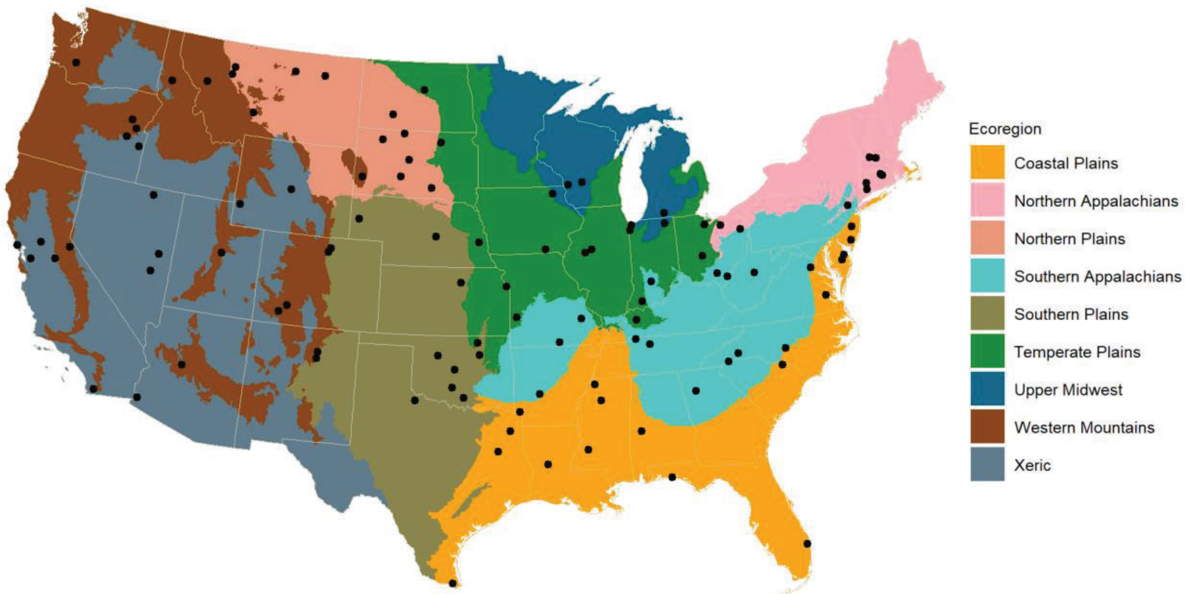


Figure 1. Location of 108 reservoirs to be sampled during SuRGE.

B1.2 Process measurement

B1.2.1 Ebullition rates

We will measure ebullition rates during the survey using inverted funnels deployed from a buoy at all sites less than 80 feet deep (floating chamber will still be deployed at these sites). The funnels intercept rising gas bubbles which collect in a reservoir attached to the top of the funnel. After a minimum 12-hour deployment period, the volume of gas in the trap will be measured and subsampled for analysis of methane, carbon dioxide, nitrous oxide, oxygen, nitrogen, and argon. See SOP# J-WECD-WMB-SOP-3948-1 'Measurement of Ebullition Rates using Passive Gas Traps' and accompanying demonstration videos ('Passive Gas Trap Deployment.mp4' and 'Passive Gas Trap Recovery.mp4'). The SOP is available at the ORD intranet site. The SOP and videos are also available at the SuRGE documents library at SharePoint.

B1.2.2 Diffusive emissions via floating chambers

Diffusive emission rates will be measured at each site using a floating chamber (Figure 2) interfaced to a portable greenhouse gas analyzer. See the demonstration videos ('Chamber Deployment.mp4', 'GGA Calibration.mp4') at the SuRGE documents library at SharePoint.

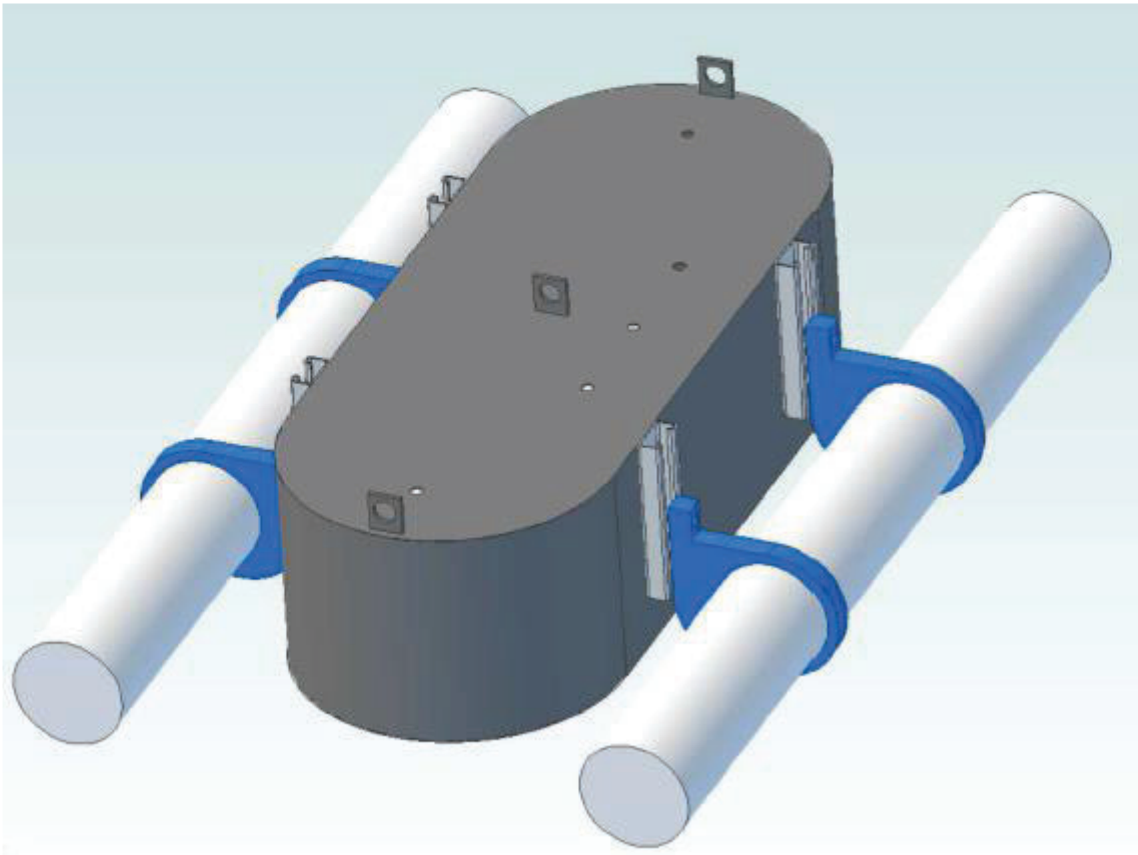


Figure 2. Three-dimensional drawing of floating chamber that will be used for measuring diffusive emission rates.

B1.2.3 Sonde measurements

Water temperature, specific conductivity, dissolved oxygen, pH, turbidity, phycocyanin, and chlorophyll will be measured using a multiparameter sonde ~0.1m below the water surface at all sites, 0.5m above the sediments at sampling sites >1 and < 4 m deep, and 1 m above the sediments at sites >= 4 m deep. The same variables will be measured throughout the water column at the Index Site (B2.4 Sonde measurements).

B1.2.4 Water chemistry and algae indicators

Water chemistry and algae indicators will be collected at the Index Site. Both sample types will be collected from a depth of ~ 0.1 m. Water chemistry will also be collected from the bottom waters at sites > 1m deep using a Van Dorn bottle, or equivalent. Water chemistry samples will be analyzed for SUVA (Specific UV absorbance), dissolved nutrients (nitrite + nitrate ($NO_{2.3}$), ammonium (NH_4^+), and reactive phosphorus (DRP)), dissolved metals, dissolved organic carbon

(DOC), dissolved anions, total organic carbon (TOC), total phosphorus (TP), and total nitrogen (TN). Algae indicators are chlorophyll a, phycocyanin, microcystin, taxonomy, and physiology.

B1.2.5 Dissolved gas samples

Triplicate dissolved gas samples will be collected from 0.1m below the water surface at the Index Site and 0.5 m above the sediments at Index sites < 4 m deep or 1 m above the sediments at Index sites >= 4 m deep. Sampling procedure will follow SOP SuRGE Dissolved Gas Sampling (ID #J-WECD-WMB-SOP-3948-1) as demonstrated in `Deep Dissolved Gas Sampling.mp4` and `Dissolved Gas Sampling.mp4`. SOP and videos are available at the SuRGE documents library at SharePoint. Gas samples will be analyzed for CH_4, N_2O, and CO_2.

B1.2.6 Air samples

Triplicate air samples will be collected approximately 2m above the air-water interface at the Index site per SOP SuRGE Dissolved Gas Sampling (ID #J-WECD-WMB-SOP-3948-1) and `Air Sampling.mp4` available at the SuRGE documents library at SharePoint.

B1.3 Photos

Field crews are encouraged to collect photos that could be useful for reconstructing conditions during the survey including weather conditions (e.g., rainy, cloudy, sunny), shoreline development patterns (e.g., forested shoreline or crowded with vacation homes), and water quality (e.g., clear water or green with algae!). Field crews are required to take two photos at the Index Site. The first photo will be of the passive gas trap deployed in the lake. This image should be taken with the boat positioned within 10 feet of the buoy and include the passive trap buoy. This image will provide an indication of water clarity. The second image is intended to give some sense of the size of the waterbody and shoreline conditions. It will be taken at the Index Site with camera pointed along the longest stretch of water unbroken by the shoreline.

B1.4 General approach and test conditions

GHG emission rates are likely to vary in response to numerous drivers including extreme water levels (high or low), rapidly changing water levels, and unusually high inflow rates. This variability is real and should be reflected in our measurements. Therefore, field crews should be prepared to sample across a broad range of conditions, unless the conditions affect safety (e.g., lightning storms) or accessibility (e.g., reservoir is dry due to extreme drought).

The sampling activities can be distributed across two days at the discretion of the Field Crew Technical Lead. However, the following groups of samples must be collected at the same time from any one particular site:

- Group 1: floating chamber measurements, air samples, shallow water temperature, and shallow dissolved gases.
- Group 2: algal indicators (chlorophyll a, phycocyanin, and microcystin).
- Group 3: deep and shallow sonde measurements

The remaining measurements can be distributed across days 1 and 2 at the discretion of the Field Crew Technical Lead. Example work distributions include:

- Example 1
    - Day 1: deploy passive gas traps, floating chamber measurements, deep and shallow sonde measurements, water chemistry, gases, algal indicators, Index Site depth profile
    - Day 2: retrieve passive gas traps
- Example 2
    - Day 1: deploy passive gas traps, floating chamber measurements, deep and shallow sonde measurements, shallow dissolved gases, and air
    - Day 2: retrieve passive gas traps, deep and shallow water chemistry, deep dissolved gases, algal indicators, Index Site depth profile
- Example 3
    - Day 1: deploy gas traps, deep and shallow water chemistry, algal indicators, deep dissolved gas, Index Site depth profile
    - Day 2: retrieve passive gas traps, floating chamber measurements, deep and shallow sonde measurements, shallow dissolved gas, air samples
- Example 4
    - Day 1: deploy passive gas traps, deep and shallow sonde measurements, Index Site depth profile, deep and shallow water chemistry, algal indicators, deep dissolved gas
    - Day 2: retrieve passive gas traps, floating chamber measurements, shallow water temperature (see F3. SuRGE GREENHOUSE GAS ANALYZER AND GPS DATA SHEET), shallow dissolved gas, air samples

B2. Sampling Methods

B2.1 Site specific factors

B2.1.1 Lake evaluation

Field Crew Technical Leads will be provided a list of Target and Oversample lakes. Prior to sampling, the Field Crew Technical Lead must determine if it is logistically possible to sample the Target lakes. This evaluation will be based on factors including accessibility (e.g., can the lake be reached by road, can a boat be launched in the lake), property rights (e.g., landowner denial), and lake-specific restrictions (e.g., no boats allowed on drinking water reservoirs). If a Target lake cannot be sampled, it must be replaced with the first Oversample lake within the same stratum. If no Oversample lakes are available for the stratum, choose from a different stratum within the same ecoregion.

Lake evaluation status must be recorded at "SuRGE Survey of Reservoir Greenhouse gas Emissions - Documents\surgeDsn\SuRGE_design_20191206_eval_status.xlsx" at the SharePoint site. Choices for Eval Status Codes include ‘LD’ (landowner denial), ‘PI’ (physically inaccessible;

no road access, no boat ramp, or no water due to dam maintenance), 'TR' (too remote, extreme effort required), and 'S' (sampleable, no barrier to site access).

B2.1.2 Locating sample sites and recording GPS tracklog

Lake specific survey design maps are available at the SuRGE group on EPA's GeoPlatform. The preferred method for using the maps to locate sampling stations is described in F16. GeoPlatform and Explorer Workflow. GPS coordinates should be recorded continuously during floating chamber deployments per F15. Bad Elf GPS USER GUIDE.

B2.1.3 Sample site evaluation

Sampling locations will be provided via the survey design maps (F16. GeoPlatform and Explorer Workflow) and a .html file summarizing survey designs for each lake (available in the lakeDsn folder at the shared documents library at SharePoint). For each sampling location an Evaluation Code must be recorded in F1. SuRGE SAMPLING EVENT SUMMARY. Acceptable entries for the Evaluation Code are TS (Target Sampled), PI (Physically inaccessible), NT (Non-Target), and NS (Target Not Sampled). TS applies to sites where no problem is encountered and the sampling proceeds as normal. Examples of PI include sites that are too shallow to access, are roped off (as is often done near dams), or a low bridge prevents access. NT applies to sites that are not within the reservoir. This can occur when the GIS shapefile isn't accurate, typically near the shorelines. NS applies to target sites that were not sampled. This might occur if the field crew runs out of time to complete the sampling or if there were problems with the sampling equipment (e.g., vandalized funnels or malfunctioning portable greenhouse gas analyzer).

Sites that have an evaluation code of PI or NT must be replaced with sites from the Oversample list. The Oversample list is a list of extra sites that can be used to replace target sites, while preserving the statistical integrity of the survey. Oversample sites must be drawn in order from the top to the bottom of the list. If the lake survey design includes sections, then each section will have a unique oversample list. This ensures that Oversample sites, when needed, are within a reasonable distance of the target site that is being replaced.

B2.2 Ebullition rates and gas composition

Ebullition rates will be measured at sites < 80 feet deep using overnight passive gas trap deployments (> 12 hours) as described in SOP# J-WECD-WMB-SOP-3948-1 'Measurement of Ebullition Rates using Passive Gas Traps' and demonstrated in 'Passive Gas Trap Deployment.mp4' and `Passive Gas Trap Recovery.mp4`. The SOP is available at the ORD intranet site and in the SuRGE documents library at SharePoint. The videos are also in SharePoint. See F2. SuRGE GAS TRAP DATA SHEET for data sheet.

B2.3 Diffusive emissions via floating chambers

See the demonstration videos (‘Chamber Deployment.mp4’, ‘GGA Calibration.mp4’) at the SuRGE documents library at SharePoint.

Methane and carbon dioxide emission rates will be measured using a version of the floating chamber technique [*Livingston and Hutchinson*]. This method consists of trapping a parcel of air between the surface of the lake and a floating chamber and monitoring how rapidly the composition of the headspace gas changes.

Headspace gas composition will be monitored in real time using a portable greenhouse gas analyzer which will continuously recirculate the chamber headspace and measure CH_4 and CO_2 concentrations every 10 seconds and store the data on an internal solid state hard drive. The Cincinnati and RTP field crews may use a Picarro G2508, Los Gatos UGGA, or Los Gatos MGGA. All other field crews will use a Los Gatos instrument. The Picarro instrument also records nitrous oxide (N_2O) concentrations.

When the portable greenhouse gas analyzer is turned on, the performance specifications in Table 2 should be checked against the instrument readout. See user manuals at the SuRGE documents library at SharePoint for operational details.

Measurement	Plausible values
LGR GGA specs	
Cell temperature	Near ambient (15 – 30C)
Ambient CH_4	1.8 – 3.0 ppm
Ambient CO_2	350 – 450 ppm
MGGA cell pressure	500 (495 – 505) torr
UGGA cell pressure	140 (138.6 – 141.4) torr
Picarro G2508 specs	
Ambient N_2O	300 – 320 ppb
Ambient CH_4	1.8 – 3.0 ppm
Ambient CO_2	350 – 450 ppm
N_2O precision	TBD
CO_2 precision	TBD
CH_4 precision	TBD
Measurement Interval	2-6 (1 point / 2-6 seconds)

Table 2. Performance specifications for greenhouse gas analyzers.

The floating chamber will consist of an aluminum chamber (20 cm height x 74 cm length x 30 cm width; Figure 2) equipped with removable floats, a small fan for mixing the chamber headspace, two ¼” Swagelok sampling ports, and a third ¼” Swagelok port interfaced to vent tubing. The sampling ports will be interfaced to 1 m lengths of polytetrafluoroethylene tubing (1/4" O.D.) connected to the portable greenhouse gas analyzer.

The floating chamber will be carefully lowered to the water surface and deployed for a minimum of 2 minutes at each site. The boat should be allowed to drift during deployment and the location recorded continuously with the provided GPS unit (or equivalent; see F15. Bad Elf GPS USER GUIDE). Record the water line relative to the graduation marks scribed on both ends of the chamber (F3. SuRGE GREENHOUSE GAS ANALYZER AND GPS DATA SHEET). These measurements will be used to estimate the volume of the chamber headspace. The gas analyzer data will be viewed in real-time via a ruggedized monitor hardwired to analyzer (Picarro) or a tablet/computer interfaced to the LGR via a wireless connection. The field analyst will immediately preview the data to ensure that an acceptable emission profile was captured. An acceptable emission profile will be characterized by CH_4 and CO_2 concentrations that smoothly increase or decrease for at least 2 minutes. If the chamber happens to capture a rising bubble, the constant rate of change will be interrupted by an abrupt increase in CH_4 concentration. Increases of < 0.5ppm CH_4 can be ignored, but the chamber will need to be vented and the deployment repeated if the concentration jump exceeds 0.5 ppm CH_4. This includes moving the boat back to the sampling site and allowing CH_4 and CO_2 concentrations to return to background levels.

Methane emission profiles characterized by concentration measurements that exhibit no temporal trend are also unacceptable. This can occur is the chamber headspace is not well mixed or the chamber is leaking. It is plausible for CO_2 to exhibit a flat concentration when dissolved CO_2 and O_2 are near equilibrium. The CO_2 profile should show increasing concentrations if dissolved oxygen (DO) is undersaturated and falling concentrations if DO is undersaturated.

B2.4 Sonde measurements

Water temperature, specific conductivity, dissolved oxygen, pH, turbidity, phycocyanin, and chlorophyll will be measured using a multiparameter sonde 0.1 m below the water surface at all sites and 0.5 m above the sediments at sampling sites between 1 and 4 m deep and 1 m above the sediments at sites >= 4 m deep (Table 3).

Site Depth	Depth for 'deep' water chemistry samples and sonde measurements
<=1 m	No 'deep' measurement
>1 m and <4 m	0.5 m above sediment
>=4 m	1.0 m above sediment

Table 3. Sample depths for deep water chemistry samples and sonde measurements.

A full depth profile will be measured at the Index Site. The shallowest measurement will always be 0.1 m below the water surface. Subsequent measurement intervals are based on site depth according to Table 4.

Index Site Depth	Measurement interval
<4m	Record measurements beginning just below the surface and at 0.5 m intervals, until 0.5 m above the bottom.
>=4m & <= 20m	Record measurements beginning just below the surface and then at 1.0 m intervals until reaching 1 m above the bottom.
>20m	Record measurements beginning just below the surface, then at 1.0 m intervals until you reach 20 m, then at 2.0 m intervals until 1.0 m above the bottom.

Table 4. Index Site depth profile measurement intervals.

Measurements will be made by lowering the sonde to the desired depth and allowing the values to stabilize. Values are typically stable when they no longer show directional drift. Water temperature typically stabilizes first with values varying by no more than ±0.1 ^{o}C. Other parameters may take longer to stabilize. Index Site depth profiles will be recorded in Appendix F4. SuRGE SONDE DATA SHEET FOR INDEX SITE DEPTH PROFILE. All other sonde measurements will be recorded in Appendix F5. SuRGE SONDE DATA SHEET FOR DEEP AND SHALLOW MEASUREMENTS

B2.5 Water chemistry and algal indicators

Water chemistry samples will be collected for the analysis of Specific UV Absorbance (SUVA), dissolved nutrients, total nutrients, total organic carbon (TOC), dissolved organic carbon (DOC), dissolved anions, and dissolved metals. Algal indicator samples will be collected for the analysis of chlorophyll *a*, phycocyanin, microcystin, taxonomy, and physiology. See Table 5 for a summary.

B2.5.1 Sample site and depths

Water chemistry and algal indicator samples will be collected at the Index Site. Both sample types will be collected from a depth of ~ 0.1 m. Water chemistry will also be collected from the bottom waters using a Van Dorn bottle, or equivalent. See Table 3 for sample collection depth.

B2.5.2 Water chemistry and algal indicator sampling

Water chemistry and algal indicator samples will be collected following the protocol described below and in depicted in Figure 4. Samples can be filtered using the provided reusable housing for 47mm filters. The reusable housing will be shipped clean and pre-loaded with 0.45um filters (polyethersulfone membrane, Fisher HPWP04700). The housing must be acid washed between uses. The disposeable 25mm syringe filters (polypropylene membrane, Fisher 6992-2504) provided in Site and QA.QC kits can also be used, but these filters will clog more quickly than the 47mm filters. Field crews may use other approaches to filter samples (e.g. Geopump and geofilter cartridge) in consultation with the Project Technical Lead. The proposed filter must be evaluated for leaching and binding of the analytes of interest.

1. **Shallow** dissolved nutrients, dissolved metals, dissolved anions, SUVA, and dissolved organic carbon.
    a. Rinse an acid washed 140 mL syringe three times with water collected from just below the water surface, avoiding any surface scum.
    b. Pull 140 mL of water into syringe and attach a preloaded filter housing.
    c. Rinse the filter by pushing ≥30 mL of water through filter. The filtrate should be discarded.
    d. Rinse the prelabeled 30 mL dissolved nutrient, 60 mL dissolved metals, 30 mL SUVA, and 30 mL dissolved anions bottles with ~5 mL of filtered site water. Repeat three times. The dissolved metals sample will be acidified in Cincinnati lab. **DO NOT** rinse the pre-acidified DOC vial.
    e. Fill the prelabeled 30 mL dissolved nutrient, 60 mL dissolved metals, 30 mL SUVA, and 30 mL dissolved anions bottles to below the shoulder with filtered site water.
    f. Fill the DOC vial completely with filtered site water.
    g. Place the samples on ice.
2. **Shallow** total nutrients, total organic carbon, and algal indicators.
    a. Rinse the two prelabeled 30 mL total nutrient vials, 20 mL microcystin vial, 1 L chlorophyll bottle, 1 L phycocyanin bottle, 30 mL algal physiology centrifuge tubes, and 250 mL algal taxonomy bottle with site water three times. **DO NOT** rinse pre-acidified TOC vial.
    b. Fill all bottles to just below the shoulder with site water <u>except</u> the 20 mL microcystin vial and the 40 mL TOC vial.
    c. Fill the 20 mL microcystin vial to ~2/3 total volume with site water from phycocyanin bottle. It is important to leave room for expansion due to freezing.
    d. Fill the pre-acidified and prelabeled 40 mL TOC vial with site water from the 1L chlorophyll bottle. Cap the vials, ensuring no headspace.
    e. Use the provided pipette (in Site kit) to add Lugol's Iodine solution to the taxonomy bottle in dropwise fashion until the water reaches the color of 'weak tea' (Figure 3). Secure lid with parafilm and place sample in zip lock bag. Lugol's iodine is relatively non-toxic, however it will stain exposed skin or clothing, so gloves are always a good idea when handling.

Figure 3. Bottle with the green star has the correct amount of Lugol's. The two with red x have too much or too little Lugol's.

f. Place all samples on ice.

3. **Deep** dissolved nutrients, dissolved metals, dissolved organic carbon, dissolved anions, total nutrients, and total organic carbon.
    a. Bottom water samples are collected using a Van Dorn bottle, or equivalent.
        i. Lower Van Dorn bottle, or equivalent, to the sampling depth (Table 3).
            1. If using a vertical sampler, drop the messenger and retrieve bottle.
            2. If using a horizontal sampler, displace the bottle horizontally ~2 m to capture an undisturbed portion of the water column. Drop the messenger and retrieve the bottle.
    b. Process the samples per steps 1 and 2 above, except that the water is collected from the Van Dorn bottle, or equivalent. The same syringe and filter can be used for both depths, provided steps 1a-d above are followed and the shallow samples are collected before the deep samples.
    c. Place all samples on ice.

B2.5.3 Chlorophyll a and phycocyanin sample processing

The chlorophyll a and phycocyanin water sample must be filtered and frozen within 24 hours. Homogenize the sample (i.e., shake, not stir) and vacuum filter in 50 mL aliquots across a 0.7µm glass fiber filter until 800 mL has been filtered or the filter becomes clogged. The vacuum pressure must not exceed 6 in. Hg (20 kPa) during filtration. Use squeeze bottle (B8.2 Base kit, F8. SuRGE EQUIPMENT AND SUPPLIES) and deionized water (B8.5 Field Crew Supplied Items; F8. SuRGE EQUIPMENT AND SUPPLIES) to rinse particles clinging to the walls of the graduated cylinder and filter tower onto the filter. Be certain to record the total volume of water filtered (excluding deionized water) in Appendix F6. SuRGE CHLOROPHYLL a/PHYCOCYANIN SAMPLE PREP DATA SHEET. Use a tweezer to fold the filter in half with the filtrate on the inside, then wrap the filter in foil, place in specimen bag, and freeze in standard commercial freezer until filters are shipped.

The 1L water sample bottles for chlorophyll a and phycocyanin may be re-used between sites on a single sampling outing. Wash each bottle out with copious amounts of tap water (e.g., hotel sink) after each sample collection. Prior to sample collection, fill, shake and empty bottles three times prior to collecting sample in the field. Upon returning from the field, wash bottles with laboratory detergent prior to next sampling.

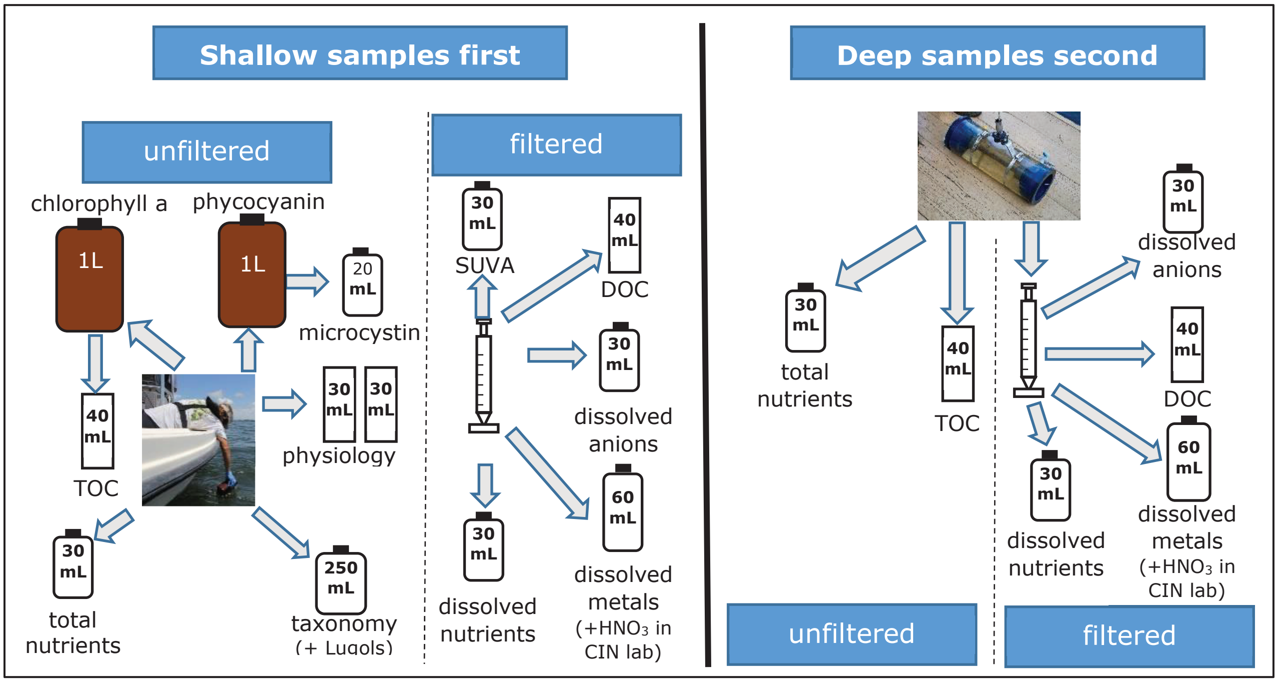


Figure 4. Flow chart for processing water samples

B2.6 Gases

B2.6.1 Dissolved gases

Triplicate dissolved gas samples will be collected from the minimum depth required to keep syringe stopcock submerged during sampling (~0.1 m), and from bottom waters (see Table 3 for sampling depth) at the Index Site. Samples will be collected in 140 mL syringes following SOP# J-WECD-WMB-SOP-3948-1 'SuRGE Dissolved Gas Sampling' available at the ORD intranet, SuRGE documents library at SharePoint, and as demonstrated in `Deep Dissolved Gas Sampling.mp4` and `Dissolved Gas Sampling.mp4` at SharePoint. Record the sampling details in Appendix F7. SuRGE DISSOLVED GAS AND AIR DATA SHEET.

B2.6.2 Air samples

Triplicate 20 mL air samples will be collected from ~ 2m above the water surface at the Index Site using a 30 mL syringe per SOP# J-WECD-WMB-SOP-3948-1 'SuRGE Dissolved Gas Sampling' available at the ORD intranet site and in the SuRGE documents library at SharePoint. The sampling methodology is demonstrated at `Air Sampling.mp4` at SharePoint. Air temperature should also be measured. Record the sampling details in Appendix F7. SuRGE DISSOLVED GAS AND AIR DATA SHEET

B2.7 Sample containers and quantities

See section B5. Quality Control for details on numbers of QA/QC samples.

Analyte Group	**Analyte**	***Filtered or unfiltered**	**Container and volume**	**Total number of samples**	**Preservation**	**Holding times**
Water chemistry	Dissolved Nutrients • $NO_{2.3}$ • NH_4^+ • reactive P	Filtered	30 mL HDPE	2 per lake + QA/QC Total = ~240	24 hours @ 5°C or 28 days at -20°C	
	Total Nutrients • TP • TN	Unfiltered	30 mL HDPE	2 per lake + QA/QC Total = ~240	24 hours @ 5°C or 28 days at -20°C	
	TOC	Unfiltered	40mL TOC vial	2 per lake + QA/QC Total = ~240	4°C + 2 drops 50% HCl (pre-acidified vials)	28 days
	DOC	Filtered	40mL TOC vial	2 per lake + QA/QC Total = ~240	4°C + 2 drops 50% HCl (pre-acidified vials)	28 days
	Dissolved metals	Filtered	60 mL HDPE bottle	2 per lake + QA/QC Total = ~240	3 drops 2% HNO_3 in CIN lab	6 months
	Dissolved anions		30 mL HDPE	2 per lake + QA/QC Total = ~240	5°C	28 days
	Specific UV Absorbance (SUVA)					Up to10 days. Preferably within 48 hours.
Gases	Dissolved N_2O, CO_2, CH_4	12mL glass vial		6 per lake Total = 648	NA, room temp	4 months
	Gas trap N_2O, CO_2, CH_4, N_2, O_2, Ar			≤45 per lake Total ≤ 4860		
	Air N_2O, CO_2, CH_4			3 per lake Total = 324		

Algae indicators	Chlorophyll a	• Water collected in opaque 1L HDPE bottle per analyte. • Filtrate collected on Glass Fiber filter. • Extract stored in glass vial		1 per lake + QA/QC Total = ~130	• Water stored on ice and filtered within ≤24 hours. • Filters stored in commercial freezers or in ice filled coolers for 10 days or less (i.e. to allow time for shipping) • Upon receipt of filters by ACESD, filters stored at -20C for ≤60 days.	
	phycocyanin			1 per lake + QA/QC Total = ~130		
	microcystin	Unfiltered	20 mL amber glass vial	1 per lake + QA/QC Total = ~130	Less than 14 days @ 4°C or several months at -20°C	
	taxonomy	Unfiltered	250mL HDPE wide-mouth bottles.	1 per lake, 83	Lugol's iodine addition in field. Months to years in dark at 4 °C. Do not freeze. Parafilm included in the sampling kit will be applied around the lid after collection.	
	physiology	Unfiltered	30mL PP centrifuge tube	2 per lake, 166	No preservation, preferably in dark at 4 °C. Do not freeze. Days to weeks.	
Sonde	Temperature	NA, *in situ*		NA, *in situ*	NA, *in situ*	NA, *in situ*
	Conductivity	NA, *in situ*		NA, *in situ*	NA, *in situ*	NA, *in situ*
	pH	NA, *in situ*		NA, *in situ*	NA, *in situ*	NA, *in situ*
	Dissolved oxygen	NA, *in situ*		NA, *in situ*	NA, *in situ*	NA, *in situ*
	Chlorophyll	NA, *in situ*		NA, *in situ*	NA, *in situ*	NA, *in situ*

	Turbidity	NA, *in situ*	NA, *in situ*	NA, *in situ*	NA, *in situ*
*Water chemistry samples can be filtered using the provided 47mm diameter x 0.45µm pore size polyethersulfone membrane filter (Fisher HPWP04700) or the provided disposable 25mm diameter x 0.45µm pore size polypropylene membrane filter (Fisher 6992-2504). Consult with Project Technical Lead before using other membrane materials. *Water for phycocyanin and chlorophyll samples will be filtered across a 47mm diameter x 0.7µm pore size glass fiber filter (Fisher 1825-047).					

Table 5. Sample containers and quantities assuming field duplicates will be collected for 10% of the unknowns.

B3. Sample Handling and Custody

B3.1 Sample labels

Site Kits and QA/QC kits (see F8. SuRGE EQUIPMENT AND SUPPLIES) will contain pre-labeled bottles for water chemistry and algal indicators (Figure 5). Chlorophyll a and phycocyanin filters will be identified by 1) a label attached to the zip lock bags containing the filters, and 2) a label printed on rite-in-rain and included in the zip lock bag with the filter. Unique samples will be identified by:

- analyte (Table 5):
  - 'dissolved nutrients', 'dissolved metals', `SUVA`, 'TOC', 'DOC', 'total nutrients', 'chlorophyll a', 'phycocyanin', 'microcystin', 'taxonomy', 'physiology'.
- QA/QC: 'duplicate' or 'blank' (QA/QC kit only)
- lake siteID: **to be populated by field crew**.
  - 7 digit alphanumeric code (i.e. ch4_009) from siteID field in survey design file. Design file available in web map, the SuRGE GeoPlatform group, and the SuRGE documents library in SharePoint.
- Station siteID: **to be populated by field crew.**
  - alphanumeric code from lake specific survey design – water chemistry and algal indicators always collected from Index site
- Date: **to be populated by field crew.**
  - mm/dd/yy format
- depth: 'shallow' or 'deep'.
- Owner: Beaulieu 513-569-7842
- Matrix: "water", "filtered solids"
- Hazards: "nonhazardous" or "weak acid"
- Preservation: "-20c", "3 drops 2% HNO3", "2 drops 50% HCl and 4c", etc.

Field crews must populate 'station siteID', 'Lake siteID', and 'Date' fields on sample labels.

Gas sample vials provided in Base Kit (see F8. SuRGE EQUIPMENT AND SUPPLIES) will be pre-labeled with an alphanumeric code containing three ID fields (Figure 5C):

- a two digit letter code identifying the project
- a two digit numeric code identifying the year
- a four digit numeric code unique to each vial

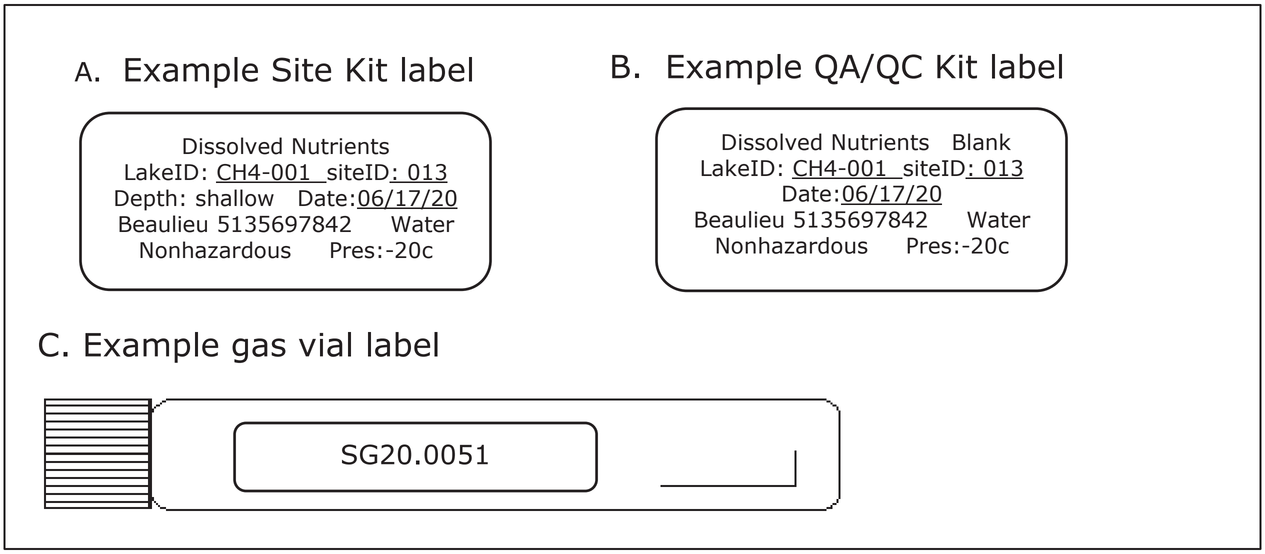


Figure 5. Example sample labels

B.3.2 Sample Tracking Sheets and Chain of Custody Forms

Sample tracking sheets found in sections F9. SuRGE WATER CHEMISTRY TRACKING SHEET,

RECEIVED FOR ANALYSIS					
Name:		Signature:		Date:	

F10. SuRGE CHLOROPHYLL, PHYCOCYANIN, AND MICROCYSTIN TRACKING SHEET, F11. SuRGE ALGAL TAXONOMY AND PHYSIOLOGY TRACKING SHEET, F12. SuRGE GAS SAMPLE TRACKING SHEET will be completed by the Field Crew Technical Lead. These sheets will be used to document shipping/delivery of samples to analytical labs, as well as receipt of samples by analytical labs. Analytical laboratories may choose to use Chain of Custody (COC) forms, in addition to sample tracking sheets, to document incorporation of samples into analytical workflow.

Sampling tracking sheets and COC forms will be uploaded to SharePoint. Figure 6 is a high-level overview of the workflow. Please see B.3.2.1 Sample Tracking Sheets and B.3.2.2 Chain of Custody Forms for details.

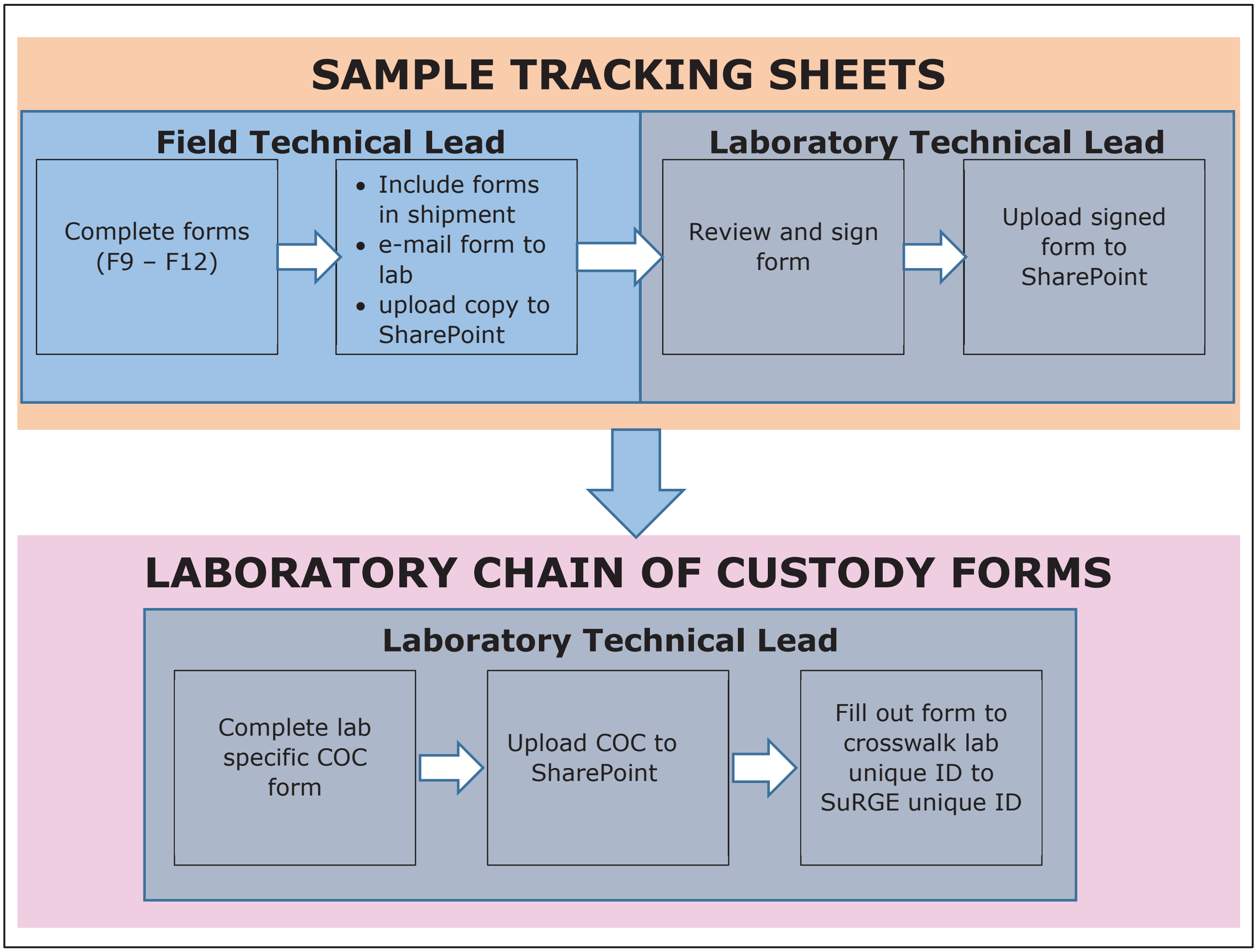


Figure 6. Workflow for Sample Tracking Forms and Chain of Custody Forms. Not all analytical analyses will use Chain of Custody forms.

B.3.2.1 Sample Tracking Sheets

Sample tracking sheets will be populated for all water chemistry, algal indicator, and gas samples submitted for analysis (F9. SuRGE WATER CHEMISTRY TRACKING SHEET,

RECEIVED FOR ANALYSIS					
Name:		Signature:		Date:	

F10. SuRGE CHLOROPHYLL, PHYCOCYANIN, AND MICROCYSTIN TRACKING SHEET, F11. SuRGE ALGAL TAXONOMY AND PHYSIOLOGY TRACKING SHEET, F12. SuRGE GAS SAMPLE TRACKING SHEET). Separate Water Chemistry and Algal Indicator sample tracking sheets will be populated for each lake, but gas samples from multiple lakes can be aggregated on one Gas Sample tracking sheet.

B.3.2.1.1 Responsibility of Sender

A hardcopy of the sample tracking sheet (in a waterproof bag or plastic sleeve) will be shipped with the samples, an electronic version e-mailed to the laboratory Technical Lead and uploaded to the 'shippingNotification' folder of the appropriate sub folder in `…/data/sampleTrackingSheets/…` at the SuRGE shared documents library at SharePoint. Files will be named by concatenating 'analyte', `lake_id`, 'shipping Lab', 'receiving lab', and 'shipping date' into a file name per Table 6. For example, the Cincinnati field crew sending algal taxonomy and physiology samples from lake 238 to Gulf Breeze on August 25, 2021 would save a copy of F11. SuRGE ALGAL TAXONOMY AND PHYSIOLOGY TRACKING SHEET named `algae238CINtoGB25August2021.pdf`.

Field	Valid values	Concatenation order
Analyte	algae, gas, chemistry	1
Lake_ID	Three digit numeric (e.g. 238, 067)	2
Shipping Lab	CIN, ADA, NAR, RTP, DOE	3
Text string	'to'	4
Receiving Lab	CIN, NAR, GB	5
Shipping Date	ddMonthYYYY	6

Table 6. Fields and valid values to be used for sample tracking sheet file names. An example file name is algae238CINtoGB25August2021.pdf

B.3.2.1.2 Responsibility of Receiving Laboratory

Upon receipt of the samples, the Laboratory Technical Lead will check the shipment contents against the tracking sheet and note any discrepancies on the tracking sheet. The Laboratory Technical Lead will also note the condition of the shipment (e.g., samples cold and in good order, samples leaking, etc) on the tracking sheet. The Laboratory Technical Lead will the sign the tracking sheet and save a copy to the appropriate subfolder in `…/data/sampleTrackingSheets/…`. The file should be named as described above (B.3.2.1.1 Responsibility of Sender) with the word 'SIGNED' appended to end of file name. Following the example above, an acceptable file name is algae238CINtoGB25August2021SIGNED.pdf.

The Laboratory Technical Lead will maintain an excel file listing all samples received, the file name of the corresponding sample tracking form, the condition of samples upon receipt, sample collection date, and sample receipt date. The Narragansett laboratory will populate "…\data\sampleTrackingSheets\NAR algal indicator\narSampleReceiptList.xlsx" at SharePoint. The Gulf Breeze laboratory will populate "…\data\sampleTrackingSheets\GB taxonomy\taxonomyPhysiologySampleReceiptList.xlsx" at SharePoint. The Cincinnati laboratory will record the condition of the samples when received at "…\data\chemistry\ttebSampleIds.xlsx" (see below for more information regarding this file).

B.3.2.2 Chain of Custody Forms

The Cincinnati Water Chemistry Technical Leads will use Chain of Custody (COC) forms to further document delivery of samples to analytical labs. This is necessary because 1) the analytes included in F9. SuRGE WATER CHEMISTRY TRACKING SHEET will be split among at least two labs, and 2) the COC form will serve as a crosswalk between the unique SuRGE sample identifiers (i.e., lake-ID, site-ID, sample depth, qa.qc) and the numeric code used by the analytical lab to track samples. Other analytical labs may use COC forms at the discretion of the Laboratory Technical Lead.

A separate CoC from will be submitted to each analyte group submitted to TTEB. The template for the chain of custody form used to submit samples to TTEB can be found at:

…\SuRGE Survey of Reservoir Greenhouse gas Emissions - Documents\data\chemistry\tteb\TTEB\TTEB Sample Submission Template.xlsx. See Readme tab in file for instructions on populating sample information.

The COC will be populated electronically (e.g. do not print and write on form), named per the conventions defined in Table 7, and saved at: …\SuRGE Survey of Reservoir Greenhouse gas Emissions - Documents\data\chemistry\tteb\TTEB.

Table 7 Fields and valid values to be used for water chemistry chain of custody forms. An example file name is ttebMetals25August2022.pdf.

Field	Valid values
Lab name	\`tteb\` for all samples submitted to TTEB
*Analyte	Metals, TOC.DOC, Anions
Submission Date	ddMonthYYYY

*Analyte: a COC form must be completed for each analyte

The COC available at "…\data\chemistry\nutrients\Nutrients COC YYYY-MM-DD_initialsTemplate.xlsx" will be used for samples submitted to Cincinnati's lachat laboratory. See notes at bottom of the \`water (original)' tab to crosswalk between SuRGE identifiers and laboratory IDs. COCs will be populated in Excel and saved to "…\data\chemistry\nutrients\". Files will be Nutrients COC_YYYY-MM-DD_initials.xlsx where "YYYY-MM-DD" is the sample submission date and \`initials\` is the initials of the person populating the file. Results will be posted to L:\Priv\Cin\ORD\Pegasus-ESF\Lachat Data\Nutrients. See Readme tab for instructions on populating COC template.

B.3.3 Shipping

During extended field campaigns it may be difficult to maintain the algal indicator and water chemistry samples at the low temperatures required for preservation (Table 5). The default procedure for temperature sensitive samples is to either keep them on ice or in hotel room freezer/refrigerator; likely a combination of the two during extended field campaigns. The Field Crew Technical Lead may choose to ship temperature sensitive samples from the field, rather than trying to keep them cool during extended sampling trips. FIELD SHIPPING IS ENTIRELY OPTIONAL. Based on past experience, water samples will remain partially frozen throughout long field days and are unlikely to be affected by freeze/thaw cycles. The chlorophyll a and phycocyanin samples are likely to thaw, however. The Narragansett laboratory will conduct tests to determine the potential impact of freeze/thaw cycles on chlorophyll a and phycocyanin.

Field crews are responsible for the cost of shipping samples to analytical laboratories. Analytical laboratories will cover the cost of returning empty coolers.

B.3.3.1 General Guidance for Keeping Samples Cool During Shipping

Water chemistry and algal indicator samples must be kept cool during storage and transport. The Base Kit (F8. SuRGE EQUIPMENT AND SUPPLIES) includes a 30 Qt Pelican brand cooler and a 15 Qt Grizzly brand cooler. These are high quality coolers featuring multiple hasps for securing the lid, silicone gaskets to minimize leakage, and sufficient insulation to hold wet ice for several days. The 15 Qt cooler should be used for shipping algal indicator samples to Narragansett and the 30 Qt for shipping chemistry samples to Cincinnati. The laboratories will return the coolers as soon as possible. Provided the procedures below are adhered to, samples may be shipped on wet ice. The Field Technical Lead must notify Jake Beaulieu if they choose to ship on dry ice. Disposable styrofoam cooler will be provided for that purpose. The coolers included in the Base Kit cannot be used for dry ice.

Samples must be contained within sealed Ziploc bags, separate from wet ice (Figure 7). This is particularly important for chlorophyll *a* and phycocyanin filters which can become saturated with water if not separated from the ice. Samples and ice must be further contained within a sealed cooler liner (e.g., thick plastic bag). These precautions are necessary to minimize the potential for leakage during transport. Shippers will hold packages that are leaking. A hard copy of the Sample Tracking Sheet must be included in every sample shipment (enclosed in Ziploc bag, see B.3.2 Sample Tracking Sheets and Chain of Custody Forms). Finally, the cooler lid must be secured with zip ties on the hasps.

Sample shipments must be carefully coordinated with the Laboratory Technical Leads. As a rule, samples cannot be received on federal holidays or weekends. To minimize the potential for samples to arrive on the weekend, shipping is restricted to Mon – Wed. During the 2021 sampling season we had several packages shipped ‘overnight’ on Thursday that weren’t delivered until Saturday.

1. Samples contained in Ziploc bags surrounded by wet ice. Samples and ice contained within cooler liner.

2. Cooler liner twisted shut, folded over on itself, and secured with zip ties.

3. Sample tracking form enclosed in Ziploc bag and placed on top of cooler liner.

Figure 7. Illustration of proper sample packaging for shipment to analytical laboratories.

B.3.3.2 Gas samples

Gas samples must be shipped via ground to avoid possible issues associated with changes in barometric pressure during flight. Samples should be sorted by sample type (e.g., AIR, dissolved gas (DG), Trap) within the provided trays and shipped within a suitable box (i.e., no refrigeration or cooler is necessary).

Ship gas samples to:
USEPA
ATTN: Daniels
MS 585
26 Martin Luther King Dr. West
Cincinnati, OH 45268
Email: Daniels.Kit@epa.gov
Phone: 513-376-1333
Alternate: Beaulieu.jake@epa.gov

B.3.3.3 Water chemistry samples

ADA will analyze dissolved nutrients, total nutrients, DOC, TOC, and dissolved anion samples at the ADA facility (no shipping required). ADA will ship dissolved metals and SUVA to Cincinnati for analysis. All other field crews will ship SUVA, dissolved anions, dissolved nutrients, total nutrients, TOC, DOC, and dissolved metals samples to Cincinnati for analysis. Glass TOC and DOC vials will be shipped within the provided bubble wrap bags.

Ship water chemistry samples to:

USEPA
ATTN: Venkatapathy
26 Martin Luther King Dr. West
Cincinnati, OH 45268
Email: venkatapathy.raghuraman@epa.gov
Phone: 513-569-7077
Alternate: Beaulieu.jake@epa.gov

B.3.3.4 Algal indicator samples

B.3.3.4.1 Chlorophyll a, phycocyanin, and microcystin

All field crews will ship chlorophyll a, phycocyanin, and microcystin to the Narragansett laboratory. The glass microcystin vials must be wrapped in included bubble wrap during shipping.

Ship chlorophyll a, phycocyanin, and microcystin samples to:
USEPA
ATTN: Shivers
27 Tarzwell Dr.
Narragansett, RI 02882
Email: shivers.stephen@epa.gov
Phone: 401-782-9629
Alternate: hollister.jeff@epa.gov

B.3.3.4.2 Algal taxonomy and physiology

All field crews will ship algal taxonomy and physiology samples to the Gulf Breeze laboratory. Samples must be shipped via overnight delivery, but do not need to be on ice. Seal lids with electrical tape or parafilm and place samples inside Ziplock bags to capture any leakage.

Ship algal taxonomy samples to:

USEPA
ATTN: Tatters
1 Sabine Island Drive
Gulf Breeze, FL 32561
Email: tatters.avery@epa.gov
Phone: 910-393-7078
Alternate: aukamp.jessica@epa.gov

B.3.4 Paper records

Field crews will populate records of sonde calibration performance and the paper records in section F. Appendix. The Field Crew Technical Lead is responsible for 1) posting electronic scans of the paper records to the SharePoint site within 30 working days of returning to laboratory, and 2) mailing the original paper records to Jake Beaulieu by September 30.

Ship paper records to:

USEPA
ATTN: BEAULIEU
26 Martin Luther King Dr. West
Cincinnati, OH 45268
Email: Beaulieu.jake@epa.gov
Phone: 513-569-7842
Alternate: white.karenm@epa.gov

B.3.5 Electronic records

Electronic records generated during this project include greenhouse gas analyzer data, GPS data (see F15. Bad Elf GPS USER GUIDE), scans of paper records (B.3.4 Paper records), transcriptions of paper records to Excel files, and results of laboratory analysis. Field based electronic records must be transferred to sensor specific subfolders at the SharePoint site within 5 working days of returning to laboratory. Water Chemistry and Gas Lab Technical Leads are responsible for depositing analytical data into analyte specific subfolders at the SharePoint site.

It is important that folder naming conventions shown in Figure 8 are adhered to; deviations will cause problems with the code used to read the data. Separate folders will be established for each field crew (e.g., CIN, RTP, USGS, R10, ADA, NAR, DOE). Names of lake specific folders must begin with 'CH4_XXX_lake.name', where 'xxx' is the three number code for each lake. The lake name following this prefix does not need to follow a specific convention. Lake names may contain a mix of upper and lower case letters and any delimiter may be used to separate words. For example, Smith Lake could be written as 'Smith Lake', 'smith lake', 'smithLake', `smith.lake', or `smith_lake'.

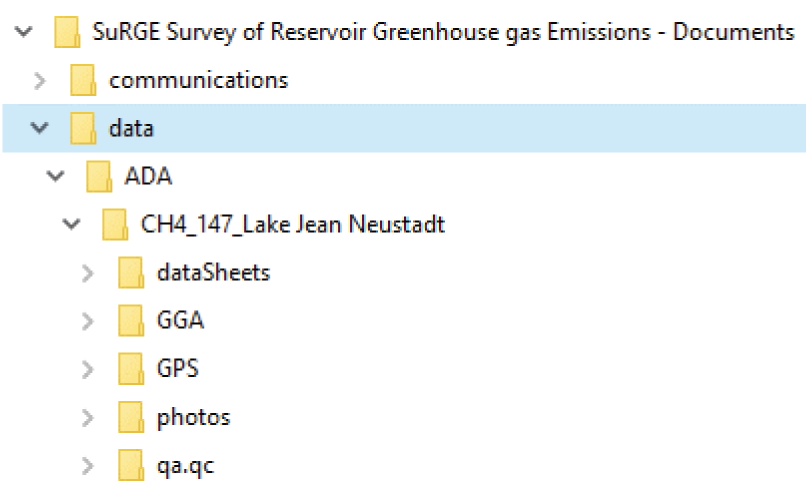


Figure 8. Directory structure of documents library at SuRGE SharePoint site.

B.3.5.1 Photos

Within five working days after returning to laboratory, the photos (B1.3 Photos) must be downloaded and transferred to the lake specific 'photos' folder at the SharePoint site. The files will be named lakexxx.jpg and trapxxx.jpg for the lake and trap images, respectively, where XXX is the three-digit numeric component of the siteID field in the SuRGE survey design file. This file can be accessed through the web app, the SuRGE GeoPlatform group, and the SuRGE documents library at SharePoint. Most cameras will generate .jpg images, but other formats (e.g., .tiff) are acceptable. Please include other photos if taken, but don't worry about renaming.

B.3.5.2 Greenhouse Gas Analyzer

The Los Gatos portable analyzers write data as .txt files which are generally downloaded as .zip files. Default file names for the Ultraportable Greenhouse Gas Analyzers (UGGA) follow "gga_YYYY-MM-DD_f####.txt" where YYYY-MM-DD is the date, and f#### is a unique serial number that counts upward to provide up to 100,000 unique file names per day. File name conventions for the Microportable Greenhouse Gas Analyzers are identical to that of the UGGA, except that 'micro' is substituted for 'gga'. Some analyzers may produce additional files, typically with the 'f' replaced with a different letter (e.g., 's', 'l', 'p', etc). It is OK to keep these files in the directory.

Ideally, one data file is generated for each field day and the file name recorded in F3. SuRGE GREENHOUSE GAS ANALYZER AND GPS DATA SHEET. Multiple files will be generated if the analyzer is shutdown/restarted during the field day. Within five working days after returning to laboratory, the data file(s) (likely a .zip file(s)) must be downloaded and transferred to the lake-specific 'GGA' folder at the SharePoint site. The .txt file should be unzipped to the folder. The file does not need to be renamed.

The Picarro G2508 will create a data file named with the following convention:

- Example: CFHADS2007-20111222-000131-DataLog_User.dat
- CFHADS: Instrument Serial Number
- 20111222: Year, month, and day of when file was started
- 000131: Hour, minute, and second of when file was started (using a 24-hour clock)

One file should be generated each day the analyzer is used, and this file name must be recorded in F3. SuRGE GREENHOUSE GAS ANALYZER AND GPS DATA SHEET.

B.3.5.3 GPS

A GPS will be used to track the boat location during the floating chamber deployment. The Base Kit will include a BadElf GPS Pro for this purpose, though field crews may use other devices at their discretion. Once data logging is complete, you can use the Bad Elf Utility app to save the recorded trip or connect the GPS to your computer with a USB cable and access the log files directly through your file explorer. See F15. Bad Elf GPS USER GUIDE

GPS data files will be named according to the following convention:
gps.sampleData.Lake (i.e., gps.03.20.2015.XXX)
where XXX is the three-digit numeric component of the siteID field in the SuRGE survey design file. If multiple files are collected on the same day, the file name can be appended with a '.1', '.2'. etc.

B.3.5.4 Field note transcription

Pegasus Technical Services, the Cincinnati on-site contractor supporting the project, will transcribe field notes into the appropriate electronic format, unless a Field Crew Technical Lead prefers to perform this work in-house.

B.3.5.4.1 Depth profiles

A separate excel file will be populated for the Index site depth profiles from each lake. The file will be based on 'surgeDepthProfileTemplate.xlsx' found in the 'data' folder at the SharePoint site. The completed template will be renamed 'surgeDepthProfilexxx.xlsx', where 'xxx' is the three digit numeric code that uniquely identifies each waterbody, and saved to the data folder specific to that waterbody (i.e., SuRGE\data\CIN\CH4_054_Spencer\dataSheets\surgeDepthProfile054.xlsx).

The 'metaData' tab of the excel file contains column descriptions. Columns are provided to flag data where there was evidence that readings were compromised. This evidence could include notes from field crews (i.e., 'pH sensor wouldn't stabilize`) or failed post deployment calibration checks.

B.3.5.4.2 Other field notes

Other field data will be populated in a copy of `surgeDataTemplate.xlsx` found in the data folder of the SuRGE SharePoint site. The file must be renamed surgeDataxxx.xlsx where `xxx` is the three-digit numeric identifier for each waterbody. The file must be stored within the 'dataSheets' folder of each lake folder.

B.3.5.4.3 Requirements for reporting chemistry results

Results from analytical laboratories must include the analytical run date. This information will be used to determine if sample hold times have been violated. Violation of sample hold times will be indicated as a value of "HOLD" in the xxxx_qual column of the final R data file. 'xxxx' is a placeholder for the analyte group (e.g., nutrients, toc.doc, metals,…).

B.3.5.5 Research Notebooks

The SuRGE documents library at SharePoint will be the central data repository. The SharePoint site contains a OneNote Research Notebook for the project (Notebook ID J-WECD-WMB-NB-2308). Edit rights to this notebook are currently limited to the Project and Field Crew Technical

Leads. Field Crew Technical Leads may choose to manage an independent Research Notebook per ORD guidance (ORD PPM 13.2).

B4. Analytical Methods

B4.1 Carbon dioxide, methane, nitrous oxide, argon, nitrogen, and oxygen

Dissolved gas samples stored in serum vials will be analyzed for CO_2, N_2O, and CH_4 via gas chromatography. Gas samples collected from the ebullition traps will be analyzed for oxygen, nitrogen, and argon, in additions to the gases identified above. All analysis will follow the latest version of SOP# J-WECD-WMB-SOP-1263.

Methane and CO_2 in the floating chamber headspace will be measured in real time using a Los Gatos Ultraportable Greenhouse Gas Analyzer or Picarro G2508. The systems use infrared detection and can make measurements as frequently as every second. Measurement precision decreases with increasing sampling frequency, however, and we will use a 5 second averaging period.

B4.2 Chlorophyll *a*

Chlorophyll *a* samples will be processed in Narragansett following the SOPs listed below available in the SuRGE documents library at SharePoint.

- SOP #J-ACESD-MAB-SOP-1425-0, *Non-Acid Determination of Chlorophyll a Using a Turner Designs Trilogy Fluorometer*
- SOP # J-ACESD-MAB-SOP-3950-0, *Chlorophyll a Standard Curve for Turner Designs Trilogy Fluorometer*

B4.3 Phycocyanin

Phycocyanin samples will be processed in Narragansett following SOP # J-ACESD-MAB-SOP-3949-0, *Determination of Phycocyanin Using a Turner Designs Trilogy Fluorometer*, available in the SuRGE documents library at SharePoint.

B4.4 Microcystin

Microcystin samples will be processed in Narragansett following SOP # J-ACESD-WEDB-SOP-443-0, *Determination of Total Microcystins Using Enzyme-Linked Immunosorbent Assay (ELISA)*, available in the SuRGE documents library at SharePoint.

B4.5 Algal taxonomy

Phytoplankton communities will be described taxonomically following SOP J-GEMMD-EDEB-SOP-4403-0, *Phytoplankton: Water sampling, cell counting, and identification*, available in the SuRGE documents library at SharePoint.

B4.6 Algal physiology

Algal physiology measurements will be made using a QAPP currently under development.

B4.7 All other analytes

The remaining analytes will be measured using standard operating procedures as detailed in Table 8.

The entire dissolved metals target analyte list for the Cincinnati lab is in Table 9. Of those analytes, six are of interest to this project and are highlighted in yellow. Any calibration and QC verification troubleshooting will be focused on those six analytes. Data for all analytes will be appropriately flagged should other analytes found in these samples be of future interest.

Analyte	**Laboratory**	**SOP ID**	**QA/QC**
TOC	MASI	WET 18	See SOP
$NO_{2.3}^-$	ADA	K-GCRD-SOP-1151-0	See SOP
NH_4^+	ADA	K-GCRD-SOP-1151-0	See SOP
Reactive P	ADA	K-GCRD-SOP-1151-0	See SOP
TOC	ADA	K-GCRD-SOP-1165-0	See SOP
TN	ADA	K-GCRD-SOP-1151-0	See SOP
TP	ADA	K-GCRD-SOP-1151-0	See SOP
$NO_{2.3}^-$	CIN	ESF-SOP-027	See SOP
NH_4^+	CIN	ESF-SOP-026	See SOP
Reactive P	CIN	ESF-SOP-029	See SOP
TOC	CIN	K-WID-SOP-3341-0	See SOP
DOC	CIN	K-WID-SOP-3341-0	See SOP
Anions	CIN	EPA Method 300.1	See SOP
TN	CIN	ESF-SOP-028	See SOP
TP	CIN	ESF-SOP-030	See SOP
Dissolved metals	CIN	SOP in development, based on EPA method 200.8	See SOP
SUVA	CIN	J-WECD-WMB-SOP-4679-0	
Temperature	NA	YSI 6600: K-LRTD-SOP-1208-0 YSI EXO: SOP in draft	See SOP
Conductivity	NA		See SOP
turbidity	NA		See SOP
pH	NA		See SOP

Analyte	Laboratory	SOP ID	QA/QC
Dissolved oxygen	NA		See SOP
Turbidity	NA		See SOP
In situ Chlorophyll	NA		See SOP

Table 8. SOPs used in this project.

DISSOLVED METALS ANALYTES (Method: EPA 200.8)	DETECTION LIMIT, µg/L
(Al) Aluminum	-0.004
(As) Arsenic	-0.004
(Ba) Barium	-0.001
(Be) Beryllium	-0.005
(CA) Calcium	-0.01
(Cd) Cadmium	-0.0003
(Cr) Chromium	-0.001
(Cu) Copper	-0.001
(Fe) Iron	-0.001
(K) Potassium	-0.3
(Li) Lithium	-0.005
(Mg) Magnesium	-0.005
(Mn) Manganese	-0.001
(Na) Sodium	-0.03
(Ni) Nickel	-0.001
(Pb) Lead	-0.002
(P) Phosphorus	-0.005
(Sb) Antimony	-0.003
(Si) Silicon	-0.02
(Sn) Tin	-0.001
(Sr) Strontium	-0.001
(S) Sulfur	-0.003
(V) Vanadium	-0.001
(Zn) Zinc	-0.0005

Table 9. Instrument dissolved metals analyte list: analytes of interest for SuRGE are highlighted.

B5. Quality Control

B5.1 Laboratory Quality Metrics

The SOPs referenced in Table 8 describe laboratory QA checks including standard curves, laboratory blanks, continuing calibration checks, and matrix spikes for lab-based measurements. Acceptance criteria and corrective actions are detailed for each SOP. Table 10 describes laboratory and field quality metrics for the greenhouse gas analyzer and the sonde measurement. See B5.2 Field Quality Metrics for more information.

Analyte	Quality metric	Frequency	Acceptance Criteria	Corrective Action
pH	post-deployment calibration check	Within 48 hours of the termination of field deployment	pH must be within 0.2 units of buffer	Recalibrate and flag field data
DO	post-deployment calibration check	Within 24 hours of the termination of field deployment	Within 5% of calculated value for water saturated air at the measured barometric pressure.	Recalibrate and flag field data
specific conductivity	post-deployment calibration check	Within 48 hours of the termination of field deployment	Within 15% of the standard value.	Recalibrate and flag field data
temperature	post-deployment calibration check	Within 48 hours of the termination of field deployment	Within 1C of value measured using a certified thermometer.	Recalibrate and flag field data
CH_4 and CO_2 via greenhouse gas analyzer	Field check with a mixed standard of 2.50 ppm and 400 ppm CH_4 and CO_2, respectively.	Once per day of field use.	Reading must within 20% of true value.	• Flag field data • Check spectra and adjust if needed • Check 10-point calibration if problem persists
	Record laser ring down times.	Once per day of field use.	No criteria but can be useful to track instrument condition.	NA
	Multipoint calibration check in the laboratory.	Beginning and end of field season.	Readings must within 15% of true value.	Recalibrate

Analyte	Quality metric	Frequency	Acceptance Criteria	Corrective Action
CH_4, CO_2, and N_2O via Picarro greenhouse gas analyzer	Multipoint calibration check in the laboratory.	Beginning and end of field season.	Readings must within 15% of true value.	Recalibrate
	Field check with a mixed standard of 2.00 ppm, 400 ppm, and 0.5 ppm CH_4, CO_2, and N_2O standard, respectively.	Once per day of field use.	Reading must within 15% of true value.	Flag field data

Table 10. QA/QC checks not specified in the SOPs reported in Table 8

B5.2 Field Quality Metrics

Field duplicates and field blanks will be collected from one reservoir during each field outing. A field outing is defined as 'continuous days in the field, without returning to laboratory'. For example, sampling three waterbodies during a continuous 1.5 week trip would be considered a single 'field outing'. Similarly, sampling one lake over two days, then returning to laboratory, would also be considered a field outing.

Portable greenhouse gas analyzer performance will be checked with a field standard during every field outing.

B5.2.1 Field blanks

Field blanks for gas samples would require a cylinder of compressed gas be transported into the field. This is beyond the scope of the project and no field blanks will be collected for gas samples.

Field blanks for water chemistry and algal indicators (excluding taxonomy and physiology) will be prepared by transferring deionized water from a clean bottle (provided in Base Kit, see F8. SuRGE EQUIPMENT AND SUPPLIES) into the appropriate bottles. Field blanks will be treated identically as the unknowns.

No analyte should be present at concentrations greater than three times the minimum detection limit in the field blanks. If this criterion isn't met, but all lab QA/QC are within range, then the field blanks were likely contaminated during sampling. The corrective action is to evaluate cleaning procedures for deficiencies.

B5.2.2 Field duplicates

Field duplicates will be collected for all algal indicators and water chemistry analytes, except algal taxonomy, algal physiology, and those measured *in situ.* All duplicates will be collected from the shallow sampling depth. These duplicates will be used to evaluate the sampling/environmental variability of the analyte concentrations. No acceptance criteria or corrective action will be associated with this quality metric.

Algal physiology samples will be collected in duplicate from all lakes. These duplicates will be used to evaluate the sampling/environmental variability of the analyte concentrations. No acceptance criteria or corrective action will be associated with this quality metric.

All dissolved gas and air samples will be collected in triplicate. Passive trap gas samples will be collected in triplicate when sufficient gas is available. These samples will be used to evaluate sampling variability for these analytes. No acceptance criteria or corrective action will be associated with this quality metric.

B5.2.3 Field standards

At the end of each field day, the portable greenhouse gas analyzer calibration should be checked with an analytical standard. This is accomplished by interfacing the instrument inlet tubing to a compressed gas cylinder via a regulator. The regulator should be set to 5 – 10 psi and the gas allowed to flow through the analyzer until the readings stabilize (approximately 1 minute). The regulator only goes up to 15 psi, which will not damage the instrument. The concentration and associated performance specifications should be recorded in F13. LOS GATOS GREENHOUSE GAS ANALYZER QA/QC SHEET or F14. PICARRO GREENHOUSE GAS ANALYZER QA/QC SHEET. Acceptance criteria for the calibration check is +/- 20% of known concentration. If the QA/QC check fails on a Los Gatos instrument, check the spectra and adjust the lasers if needed. See user manual in SuRGE documents library at SharePoint for more information.

After the measurement has stabilized and has been recorded, the instrument should be shut down while the calibration standard is flowing through the instrument. This procedure ensures that the moisture content of the measurement cell is low during instrument shut down. If, during shut down, the water vapor mixing ratio is within 20% or 5% of the saturation value (Table 11) for the LGR and Picarro, respectively, water can condense in the measurement cell and potentially affect precision and bias. Based on previous deployments, it is not uncommon for water vapor to exceed the saturation point when the chamber is floating on the water surface, though values typically fall when the chamber is pulled into the boat. Measurement cells can also be dried by passing air through an in-line desiccant column prior to being delivered to analyzer.

T (deg F)	T (deg C)	Saturation mixing ratio (ppm)	Acceptable level (ppm)	
			LGR	Picarro
32	0	6,000	4,800	5700
41	5	8,500	6,800	8075
50	10	12,100	9,680	11495
59	15	16,800	13,440	15960
68	20	23,000	18,400	21850
77	25	31,200	24,960	29640
86	30	41,800	33,440	39710
95	35	55,400	44,320	52630
104	40	72,600	58,080	68970

Table 11. Maximum H2O mixing ration for shut down of Los Gatos and Picarro analyzers as a function of ambient temperature.

B6/B7. Instrument/Equipment Calibration, Testing, Inspection, Maintenance

B7.1 Greenhouse gas analyzer

All greenhouse gas analyzers (Picarro and LGR models) will have the factory calibration tested against 10-point curve in the Cincinnati gas lab per SOP 1273-1 ‘Technical SOP for the 10-point Calibration Verification of the Los Gatos Research Ultraportable Greenhouse Gas Analyzer (LGR-UGGA)’ at the beginning and end of the field season (SOP available at ORD intranet site and SuRGE documents library at SharePoint). Per vendor recommendation, data will be corrected for any deviation from a 1:1 relationship between measured and known values, rather than changing the instrument calibration. Data correction will be performed by the Project Technical Lead.

LGR greenhouse gas analyzers are covered by warranties that include an annual ‘remote health check’ by the vendor. This preventative maintenance will be conducted near the beginning of each field season.

B7.2 Thermometers

The Base Kit will include a new digital thermometer and NIST traceable calibration certificate. The calibration certificate is only valid for one year, therefore new thermometers will be provided each year. Alternatively, the Field Crew Technical Lead may recertify the expired calibration following CESER SOP K-LRTD-SOP-1172-1, “Thermometer Calibration”. SOP available at ORD intranet site and SuRGE documents library at SharePoint.

B7.3 Barometers

Calibrations on Field Crew supplied barometers must be checked annually. Most field crews will use YSI brand barometers with factory calibration tolerance of ±1mm Hg. If the Field Crew

Technical Lead cannot check the barometer calibration locally, this service can be performed through the CEMM QA office in Cincinnati. Contact the SuRGE project QA/QC officer for details.

B7.4 Cleaning

Several durable items in the Base Kit (see F8. SuRGE EQUIPMENT AND SUPPLIES) require cleaning between uses. Items not specifically mentioned in 'B7.4.1 Water chemistry' and 'B7.4.2 Algal indicators' do not need cleaning between uses.

B7.4.1 Water chemistry

The Base Kit (see F8. SuRGE EQUIPMENT AND SUPPLIES, B8.2 Base kit) includes five syringes and housings for 47mm filters to be used for sampling dissolved nutrients, dissolved organic carbon, dissolved anions, and dissolved metals. Syringes and filter housings must be cleaned with a laboratory detergent (e.g. Alconox or equivalent), soaked in an acid bath of 5% ACS trace-element grade HCl for 30 minutes, and rinsed 3 times with DI water.

B7.4.2 Algal indicators

The Base Kit (see F8. SuRGE EQUIPMENT AND SUPPLIES, B8.2 Base kit) includes three opaque 1 L bottles for chlorophyll a sampling (i.e., one bottle for unknowns, one for blanks, and one for duplicates), three opaque 1 L bottles for phycocyanin sampling (i.e., one bottle for unknowns, one for blanks, and one for duplicates), and one filter tower assembly. During extended field trips it is sufficient to clean this equipment using tap water in the hotel (or equivalent) between uses. Equipment should be washed in a laboratory detergent (e.g. Alconox or equivalent) and rinsed with DI water upon return to laboratory.

B7.4.3 Watercraft and Equipment

The watercraft and equipment that are deployed in reservoirs may become contaminated with invasive species. A variety of preventative measures may be taken to minimize the transfer of these "hitchhikers" among reservoirs as teams move from site to site. SuRGE strongly recommends the following minimum precautions:

Clean: the trailer, boat hull, motor and equipment that were deployed will be cleaned. Use provided scrub brush and scrubbing pad to remove all visible plants, animals and mud.

Drain: all sources of stored water will be drained. Remove the boat plugs, drain all bilge, live well and motor water.

Dry: boat, motor and trailer will be dried with provided shammy towel, or equivalent, unless the boat will remain out of water for at least five days.

If possible, pressure spraying boat and equipment (e.g., manual car wash) may further ensure non-native species are removed.

Field Crew Technical Leads may optionally choose to disinfect watercraft and equipment. Per New York State guidance, SuRGE recommends one of the following disinfection methods:

- hot water greater than 120°F
- 2% bleach (10% if whirling disease is present; caution, corrosive to aluminum)
- 200ppm potassium chloride (2 teaspoons/2 gallons water).

SuRGE will include KCl in Base Kit (B8.2 Base kit; F8. SuRGE EQUIPMENT AND SUPPLIES) to be optionally used for disinfection. Mix one container of KCl with 2.5 gallons of water. Anchors and rope could be dipped in KCl solution; larger pieces of equipment (including boat hull) could be wiped with solution.

Teams should be aware of local regulations concerning watercraft and equipment regarding aquatic invasive species and the presence of invasive species in reservoirs they visit. The following are helpful resources:

https://stopaquatichitchhikers.org/

https://www.invasivespeciesinfo.gov/subject/watercraft-inspection-and-decontamination-programs

http://nsglc.olemiss.edu/projects/model-legal-framework/files/state-comparison-revised.pdf

B8. Inspection/Acceptance of Supplies and Consumables

See F8. SuRGE EQUIPMENT AND SUPPLIES for a complete equipment list. The Project Technical Lead is responsible for ensuring that supplies shipped to field crews contain the listed equipment and supplies and that the equipment and supplies are of the required quality for collection of samples and field measurement. The Technical Leads for analytical labs in Cincinnati, Ada, and Narragansett are responsible for ensuring supplies and consumables meet project quality standards. Consumables are inspected when received. Consumables with expiration dates will be used prior to expiration unless recertified or otherwise justified in research records.

B8.1 Greenhouse gas analyzer kit

USEPA/ORD/CEMM/WECD/WMB (Cincinnati) will provide each field crew with a CO_2/CH_4 analyzer and floating chamber to measure CO_2 and CH_4 diffusive emission rates. The analyzer will be either a Los Gatos UltraPortable Greenhous Gas Analyzer (UGGA) or a Los Gatos Microportable Greenhouse Gas Analyzer (MGGA). The Cincinnati field crew may also use a Picarro G2308. The Los Gatos analyzers are controlled via VNC Viewer freeware running on iOS, android, or PC. An iOS iPad will be provided with each Los Gatos instrument, but local field crews may use other devices if preferred. See F8. SuRGE EQUIPMENT AND SUPPLIES for list of kit contents.

B8.2 Base kit

The Base Kit is comprised of the subset of durable equipment and supplies needed to execute the survey. Each field crew will be supplied a Base Kit by USEPA/ORD/CEMM/WECD/WMB (Cincinnati), unless the field crew chooses to supply their own Base Kit. See F8. SuRGE EQUIPMENT AND SUPPLIES for list of kit contents.

B8.3 Site kit

A Site Kit contains the subset of consumable supplies (i.e., items that require replacement after use) and will be provided by USEPA/ORD/CEMM/WECD/WMB (Cincinnati). The site kit will contain all sample bottles necessary for sampling a single lake. Crews should consider having at least one additional site kit available as a spare should any supplies be lost. See F8. SuRGE EQUIPMENT AND SUPPLIES for list of kit contents.

B8.4 QA/QC kit

No QA/QC sample will be collected for algal taxonomy. Duplicate algal physiology samples will be collected from all lakes, therefore no algal physiology bottles will be included in QA/QC kit. One field duplicate and field blank will be collected for each water chemistry analyte (SUVA,TOC, DOC, TN/TP, NO2.3/NH4+/SRP, anions, metals), chlorophyll a, phycocyanin, and microcystin during each 'field outing'. A field outing is defined as 'continuous days in the field, without returning to laboratory'. For example, sampling three waterbodies during a continuous 1.5 week trip would be considered a single 'field outing'. Similarly, sampling one lake over two days, then returning to laboratory, would also be considered a field outing. See F8. SuRGE EQUIPMENT AND SUPPLIES for list of kit contents.

USEPA/ORD/CEMM/WECD/WMB (Cincinnati) will distribute QA/QC kits to field crews based on anticipated sampling schedule.

B8.5 Field Crew Supplied Items

The field crew will also supply items for the survey. These items include general field equipment (i.e., printed data sheets, pens), limnological equipment (i.e. multi-parameter probe), and boat equipment. F8. SuRGE EQUIPMENT AND SUPPLIES for items the field crew will need to provide.

B9. Non-direct Measurements

Data collected from a previous methane emission rate project, covered under QAPP # G-WSWRD-0030103-QP-1-0, may be used with data collected under this project. If other sources of secondary data are used, an addendum or a memo to file will be added to this QAPP in the QA Track database.

B10. Data Management

B10.1 Data analysis workflow

The SuRGE documents library at SharePoint will be the central data repository. Field Crew Technical Leads will be responsible for uploading electronic data per sections 'B.3.4 Paper records' and 'B.3.5 Electronic records'. The Project Technical Lead will be responsible for organizing a collaborative and reproducible data submission and analysis workflow. This will be accomplished by 1) using R for data analysis and visualization, 2) using a public repository under the EPA's institutional account for sharing code, and 3) using git to establish version control over code. Additional benefits of this workflow are 1) all EPA data will be accessible to collaborators (including outside collaborators), but not the general public, and 2) all electronic resources will be protected from loss via institutional back-up safeguards.

The Project Technical Lead will lead most aspects of data analysis leading to a final aggregated data set. This will include 1) writing code to read in field and laboratory data, 2) calculate derived quantities from measured data (e.g., diffusive emission rates from gga data, dissolved gas concentrations from gas chromatograph data), 3) aggregate across field duplicates, 4) track QA/QC flags, and 5) upscale point measurements to reservoir-scale estimates of mean and variance.

The final aggregated data set will be used to generate a national-scale estimate of CH_4 and CO_2 emissions from U.S. waterbodies. This effort will be conducted collaboratively with all interested SuRGE team members. We have identified several approaches for upscaling our measurements to the nation including 1) classical survey design calculations, 2) using the data train a predictive model for estimating emissions from all U.S. reservoirs, and 3) a combination of the two. Survey design calculations will be executed using function in the spsurvey library in R and features of the SuRGE probabilistic survey design. We will likely explore a variety of modeling approaches including machine learning algorithms, generalized linear models, generalized additive models.

B10.2 Laboratory specific data management

Technical Leads for the Water Chemistry and Gas labs are responsible for ensuring that reported data meet the relevant QA/QC criteria, per the SOPs, or are flagged appropriately. Data should be uploaded to SharePoint per 'B.3.5 Electronic records'. A variety of reporting formats are accepted, as long as reports are clear with respect to analytes, flags, and concentration units.

C. ASSESSMENTS AND OVERSIGHT

C1. Assessments and Response Actions

C2. Reports to Management

The Project Technical Lead will be responsible for reports to management. These will likely come in the form of informal briefings to staff in the Office of Air and Radiation, the National Program Director and associated staff in the Air and Energy national research program, and management within the Center for Environmental Measurement and Modeling.

Tangible products from this research will include an estimate of GHG emissions from US reservoirs for inclusion in the Inventory of US GHG Emissions and Sinks and at least one journal article in a peer reviewed journal.

The Project Technical Lead will organize periodic project meetings, primarily via Skype, and will look for opportunities to conduct in-person meetings. In-person meetings will most likely be attached to pre-existing meetings that SuRGE team members plan to attend such as conferences.

D. DATA VALIDATION AND USABILITY

D1/D2. Data Review, Verification, and Validation/Verification and Validation Methods

Problems with data integrity are most likely to arise when transcribing paper records to electronic format (see B.3.4 Paper records). To address this issue, Pegasus Technical Services will be responsible for ensuring that 100% of entered data is checked for accuracy. Data review will be documented using a Review Documentation Form provided by Pegasus Technical Services. The Project Technical Lead will develop additional QA/QC checks designed to detect egregious data entry issues (e.g., misplaced decimal place, incorrect units).

Data problems can also arise due to coding errors. To address this issue, SuRGE team members will be encouraged to clone the Github repository (B10.1 Data analysis workflow) and run/inspect the code. Code used for upscaling our measurements to the nation must be run by at least two team members and the results compared for inconsistencies.

D3. Analysis and Reconciliation with User Requirements

The Project Technical Lead will work closely with Office of Air and Radiation (OAR) to ensure the data are reported in a format consistent with their intended use. Formats will include a peer-reviewed manuscript describing the main project results and an emission rate estimate reported per the standardized reporting requirements for Inventory of GHG Emissions and Sinks as dictated by the United Nations. This will include a brief description of how the data were generated and aggregated for use in the inventory. OAR will submit the report for additional national and international review.

E. References

Livingston, G. P., and G. L. Hutchinson Enclosure-based measurement of trace gas exchange: applications and sources of error, in *Methods in Ecology: Biogenic trace gases: Measuring emissions from soil and water*, edited by P. A. Matson and R. C. Harriss, pp. 14-51, Blackwell Science LTD, Oxford.

F. Appendix

F1. SuRGE SAMPLING EVENT SUMMARY

Lake siteID:	________	Sampling Dates:	___________	Timezone:	_______________
INDEX siteID:	_______	*Trip ID:	___________	QA.QC lake:	☐ Yes ☐ No
Reservoir outflow present?:	☐ Yes ☐ No				

siteID	¥Eval status	siteID	¥Eval status	siteID	¥Eval status	siteID	¥Eval status

¥Eval Status: TS (Target Sampled), PI (Physically inaccessible), NT (Non-Target), NS (Target Not Sampled).
*Trip ID: lake_id for lakes included in sampling trip (e.g. 64-278-126)

General notes (weather, algae bloom, etc):

Name (print):	___________	Signature:	_________________________	Date:	___________

F2. SuRGE GAS TRAP DATA SHEET

Lake siteID:	______________________	Time zone:	_________________	Year:	______________	Exetainer code prefix:	____________

Station siteID	Lat xx.xxxxxx	Long xxx.xxxxxx	Deployment time/date hh:mm mm/dd	Retrieval time/date hh:mm mm/dd	Volume recovered (mL)	Last four digits of Exetainer codes		

Comments:

Name (print)	______________________	Signature:	______________________________	Date:	_______________

F3. SuRGE GREENHOUSE GAS ANALYZER AND GPS DATA SHEET

Lake siteID:	___
Sample Date:	___
Time zone for gas analyzer:	___
Gas analyzer file name:	___
*gps data logging turned on?	___

*GPS unit will collect positional data throughout all chamber deployments

Station siteID	Chamber start time (from analyzer clock)	⁺Water temperature (^oC)	Chamber volume graduation		*CH_4 and CO_2 profile checked?	
					YES☐	No☐
					YES☐	No☐
					YES☐	No☐
					YES☐	No☐
					YES☐	No☐
					YES☐	No☐
					YES☐	No☐
					YES☐	No☐
					YES☐	No☐
					YES☐	No☐
					YES☐	No☐
					YES☐	No☐
					YES☐	No☐
					YES☐	No☐
					YES☐	No☐
					YES☐	No☐
					YES☐	No☐

Name (print):	___	Signature:	___	Date:	___

*CH_4 should steadily increase during chamber deployment. CO_2 may increase or decrease, depending on algae productivity.
⁺Only record water temperature if not recording ‘shallow’ sonde data.

F4. SuRGE SONDE DATA SHEET FOR INDEX SITE DEPTH PROFILE

Lake siteID: __________ Index siteID: _____________ Index site depth (m): ________ Date: __________

Depth(m) xx.x	Temp (C) xx.x	DO (mg/L) xx.x	sp.Cond (μs cm^{-1}) xx	pH xx.x	Chl (ug/L) xx.x	Phyc (ug/L) xx.x	Turbidity (NTU) xx.x	Flags	Depth(m) xx.x	Temp (C) xx.x	DO (mg/L) xx.x	sp.Cond (μs cm^{-1}) xx	pH xx.x	Chl (ug/L) xx.x	Phyc (ug/L) xx.x	Turbidity (NTU) xx.x	Flags

Flag	Comments

Flag codes: k = No measurement or observation made, U = suspect measurement or observation, F1, F2, etc = flags defined by field crew. Explain all tags in comments section.

Name (print): ____________________________ Signature: ________________________ Date: ____________

F5. SuRGE SONDE DATA SHEET FOR DEEP AND SHALLOW MEASUREMENTS

Lake siteID: ____________________ Date: ______________________

		SHALLOW MEASUREMENTS									DEEP MEASUREMENTS								
Station siteID	Site depth(m) xx.x	Sample depth(m) xx.x	Temp (C) xx.x	DO (mg/L) xx.x	sp.Cond (µs cm^{-1}) xx	pH xx.x	Chl (ug/L) xx.x	Phyc (ug/L) xx.x	Turbidity (NTU) xx.x	Flag	Sample depth(m) xx.x	Temp (C) xx.x	DO (mg/L) xx.x	sp.Cond (µs cm^{-1}) xx	pH xx.x	Chl (ug/L) xx.x	Phyc (ug/L) xx.x	Turbidity (NTU) xx.x	Flag

Flag	Comments

Flag codes: k = No measurement or observation made, U = suspect measurement or observation, F1, F2, etc = flags defined by field crew. Explain all flags in comments section.

Name (print):	______________________	Signature:	__________________	Date:	__________

F6. SuRGE CHLOROPHYLL a/PHYCOCYANIN SAMPLE PREP DATA SHEET

Collection date	Lake siteID	Station siteID	*Sample Type	⁺Analyte	Filter date	Volume filtered (mL)

*unknown (UNK), duplicate (DUP), or blank (BLK) ⁺Chlorophyll a or phycocyanin

Name (print):		Signature:		Date:	

F7. SuRGE DISSOLVED GAS AND AIR DATA SHEET

Lake siteID:	________	Index siteID:	___________	Date:	____________	Exetainer code prefix:	________
Thermometer serial number:	____________________________________						

DISSOLVED GAS SAMPLING DATA				
*Sample depth	Sample depth (m)	Last four digits of Exetainer code	Headspace equilibration temperature (C)	Notes/Comments
shallow	~0.1m			
shallow	~0.1m			
shallow	~0.1m			
deep				
deep				
deep				

*'shallow' samples collected from minimum depth required to maintain syringe stopcock under water surface. This depth could exceed 10 cm if waves are present. If site depth <1m, no deep samples are collected. If site depth is >1m and <4m, deep samples are collected from 0.5m above sediment. If >=4m deep, samples collected from 1m above sediment.

AIR SAMPLING DATA				
Barometer identifier:	____________________________________			
BP (mm Hg)	Air Temperature (C)	Last four digits of atmospheric air Exetainer odes		

Name (print):	___________________	Signature:	_______________________	Date:	_________

F8. SuRGE EQUIPMENT AND SUPPLIES

The equipment detailed below will be provided to each field crew in four separate shipments. The bulk of the materials will be shipped in a large Gaylord box mounted to a pallet. The pre-acidified TOC vials, compressed gas cylinder, and Greenhouse Gas Analyzer battery will be shipped as three separate Dangerous Goods shipments. We do not anticipate that field crews will need to return the disposable compressed gas cylinders. The TOC vials can be returned to Cincinnati via regular shipping after they have been filled with site water. The Greenhouse Gas Analyzer battery will need to be returned to Cincinnati via Dangerous Goods shipping. Please see 'HASP 2020-036 Rev.1.pdf' at SharePoint for additional guidance from Cincinnati SHEM.

Greenhouse Gas Analyzer Kit

USEPA/ORD/CEMM/WECD/WMB (Cincinnati) will provide each field crew with a portable greenhouse gas analyzer. Analyzers will be returned to Cincinnati at end of each field season.

Greenhouse Gas Analyzer Item	Quantity	Protocol
Picarro G2308, Los Gatos UGGA, or Los Gatos M-GGA	1	Diffusive emissions
Analyzer battery	1	Diffusive emissions
Battery to analyzer cable	1	Diffusive emissions
Charger for analyzer battery	1	Diffusive emissions
Male cigarette plug to analyzer power cable and 12V plug socket with clamps	1	Diffusive emissions
AC power cable for analyzer (wall outlet to analyzer)	1	Diffusive emissions
Analyzer maintenance kit	1	Diffusive emissions
Inlet filter (**UGGA only**)	1	Diffusive emissions
Barbed fittings for circulation tubing (**MGGA only**)	4	Diffusive emissions
Field standard (CH_4 and CO_2), regulator, and tubing for analyzer	1	Diffusive emissions
Float chamber, tubing, and 12V plug socket with clamps	1	Diffusive emissions
¼ Swagelok nut with front and rear ferrule	3	Diffusive emissions
Spare fan for float chamber	1	Diffusive emissions

iPad with Explorer and LGR software (VNC), AC charger base, lightning cable	1	Diffusive emissions and locating sampling sites
Bad Elf bluetooth GPS , charging cable, USB to male cigarette port adapter, AC butt, waterproof bag	1	Locating sites

Base Kit

USEPA/ORD/CEMM/WECD/WMB (Cincinnati) will provide each field crew with a compete Base Kit. Field crews will keep the Base Kit through the duration of their involvement in the project.

Base Kit Item	Quantity	Protocol
Pre-evacuated and labeled Exetainers in test tube racks (up to 69 per lake)	Dependent upon number of sites a crew plans to sample	Dissolved gas and air sampling
Scrub brush	1	Cleaning equipment
Potassium chloride (KCl)	15 2g containers	Cleaning equipment/boat
Shammy towel	2	Cleaning boat
Nut driver for hose clamps	1	Passive traps
Adjustable wrench for quick links	1	Passive traps
6" pliers	1	Passive traps
Extra quick links	5	Passive traps
Extra brass rings	5	Passive traps
Teflon tape	1 role	Passive traps
Funnels with eyelets	22	Passive traps
Tall (32") upright sets (one set = 3 rods) with 6 brass rings and 4 quick link per set.	17	Passive traps
Tall gas collection tubes	17	Passive traps
Short (15") upright sets (one set = 3 rods) with 6 brass rings and 4 quick link per set.	5	Passive traps
Short gas collection tubes	5	Passive traps
Buoys	22	Passive traps
Roll of reflective tape for buoy	1	Passive traps

Buoy labels	22	Passive traps
Rope kit • 8-10’ • 15-15’ • 3-20’ • 16-35’ • 5-40’ • 1-70’	1	Passive traps
Anchors	22	Passive traps
140 mL syringe with one-way stopcock	3	Shallow dissolved gas sampling
140 mL syringe with three-way stopcock	3	Deep dissolved gas sampling
140 mL syringe, no stopcock	1	Passive trap sampling
30 mL syringe with one-way stopcock	6	Passive trap and air sampling
Needle, 27 gauge	20	Passive trap, dissolved gases, air samples
250 mL bottle for Sharps box	1	Needle disposal
50 mL centrifuge tube	1	Dissolved gas sampling
Thermometer	1	Dissolved gas sampling
0.5 L graduated cylinder (plastic)	1	Chlorophyll/phycocyanin
Opaque 1L bottle	6	Chlorophyll/phycocyanin (2 for unknowns, 2 for blanks, 2 for duplicates)
1 L bottle	2	Field blank
500 or 1000 mL bottle	1	Water for filter tower rinse
Squeeze bottle	1	Rinsing filter tower
Acid washed 140 mL syringe for chemistry samples	5	Water chemistry
Acid washed housing for 47mm syringe filter	5	Water chemistry
47mm, 0.45um filters	4 per lake, 4 per QA/QC kit, plus ≥10 extra	Water chemistry
Nitrile gloves	1 box of S/M/L	Collecting water chemistry, processing algal indicator

Hand or battery powered vacuum pump with gauge	1	Chlorophyll
Vacuum flask	1	Chlorophyll
Filter assembly	1	Chlorophyll
Forceps	1	Chlorophyll
Aluminum foil	1 roll	Chlorophyll
Lugol's iodine aliquot (10 mL)	1	Algal taxonomy
Rite in the Rain pen	2	Data sheets
Zip Ties	500	Passive traps, etc
30 Qt cooler	1	Shipping chemistry
15 Qt cooler	1	Shipping algal indicator to Narragansett
Cooler liners	1 box	Shipping

Site Kit

The Site Kit contains consumables used at each waterbody. USEPA/ORD/CEMM/WECD/WMB (Cincinnati) will provide each field crew with one Site Kit per waterbody, based on anticipated sampling schedules.

Site Kit Item	Quantity	Protocol
Disposable syringe filter	4	Can be used to filter chemistry samples.
40 mL vial	4	Water chemistry (TOC, DOC)
20 mL amber glass vial	1	Algal indicator (microcystin)
30 mL HDPE vial	6	Water chemistry (total nutrients, dissolved nutrients, total anions)
60 mL HDPE vial	2	Water chemistry (metals)
0.7µm glass fiber filters in specimen bags	2 + 2 extra	Algal indicators (chla, phycocyanin)
250 mL HDPE narrow mouth bottles in ziplok bag with parafilm strip and pipette	1	Algal taxonomy

30 mL PP centrifuge tube and parafilm strip	2	Algal physiology

QA/QC Kit

No QA/QC sample will be collected for algal taxonomy. Algal physiology samples will be collected in duplicate from all lakes, therefore no physiology bottles will be included in QA/QC Kit. No blanks will be collected for physiology or taxonomy samples.

One field duplicate and field blank will be collected for each water chemistry analyte (TOC, DOC, TN/TP, NO2.3/NH4+/SRP, anions, metals), chlorophyll a, phycocyanin, and microcystin during each 'field outing'. A field outing is defined as 'continuous days in the field, without returning to laboratory'. For example, sampling three waterbodies during a continuous 1.5 week trip would be considered a single 'field outing'. Similarly, sampling one lake over two days, then returning to laboratory, would also be considered a field outing.

Field blanks for filtered samples must be collected using a clean filter, filter housing, and syringe.

USEPA/ORD/CEMM/WECD/WMB (Cincinnati) will distribute QA/QC kits to field crews based on anticipated sampling schedule.

QA/QC Kit Item	Quantity	Protocol
Disposable syringe filter	4	Can be used to filter chemistry samples.
40 mL TOC vial	4	Water chemistry (TOC, DOC): blank and field duplicate
20 mL amber vial	2	Algal indicator (microcystin): blank and field duplicate
30 mL HDPE vial	6	Water chemistry (total nutrients, dissolved nutrients, dissolved anions): blank and field duplicate
60 mL HDPE vial	2	Water chemistry (metals): blank and field duplicate
0.7µm glass fiber filters in specimen bags	4 + 2 extra	Algal indicators (chla, phycocyanin): blank and field duplicate

Field Crew Supplied Items

Field Crew Supplied Item	Quantity	Protocol

Multiparameter sonde with probes for optical DO, chl, pH, turbidity, temperature and conductivity. Load with fresh batteries.	1	Site physicochemical characterization
Calibration cups and standards for multiparameter sonde	1	Sonde calibration
Weighted sonde deployment cap	1	Site physicochemical characterization
Sonde communication cable. Crews may choose to procure a cable sufficiently long to conduct depth profiles. Alternatively, depth profiles may be logged internally without use of communication cable.	1	Site physicochemical characterization
Sonde display unit with barometer	1	Site physicochemical characterization
Van Dorn bottle, or similar	1	Water chemistry
Depth Finder (hand-held or boat mounted sonar)	1	Site physicochemical characterization
Cooler with ice for field use	1	Water chemistry
Two 1L bottles of laboratory grade deionized water for field blank. Bottles provided in Base kit.	1	QA/QC
500mL distilled water. Bottle provided in Base kit.	1	Algal indicator
Water resistant paper		Data sheets
acetone and methanol	15mL each	Greenhouse Gas Analyzer maintenance kit
Electronics compressed gas duster	1	Greenhouse Gas Analyzer maintenance kit
OPTIONAL: 5-gallon bucket for equipment disinfection	1	KCl solution for cleaning boat/equipment between lakes
OPTIONAL: Field laptop with Explorer and software for GHG analyzer.	1	Locating sites

OPTIONAL: Cell phone for site pictures. Can use provided iPad.	1	Site description
OPTIONAL: GPS unit	1	Site location

Boat Equipment List

Suggested boat equipment

Item
Personal Flotation Device (see local requirements)
SHEM approved boat safety bag
Boat anchor w/100 to 200 foot lines (line in a bucket or spool)
Paddle
Push pole for shallow waters
Wheel lug nut wrench, spare tire, and jack (probably use vehicle jack)
Gas and oil can
Spare prop and shear pin
Boat plug (extra)
Bow/stern lights
Fire extinguisher

F9. SuRGE WATER CHEMISTRY TRACKING SHEET

SuRGE WATER CHEMISTRY SAMPLE TRACKING SHEET

SHIPPING INFORMATION

Sender: ________________________ Sender Phone: ________________

Shipped by: ○ FedEx ○ UPS ○ Other: ________________________

Tracking Number: ________________________ Date Sent: ________________

LAKE INFORMATION

Lake siteID: ________________________ Crew: ________________

Dates Collected: ________________________

SAMPLE INFORMATION

ROUTINE SAMPLES		DUPLICATES		BLANKS	
Sample type	Collected?	Sample type	Collected?	Sample type	Collected?
Dissolved nutrients - deep	☐	Dissolved nutrients - shallow	☐	Dissolved nutrients	☐
Dissolved nutrients - shallow	☐	Dissolved metals - shallow	☐	Dissolved metals	☐
Dissolved metals - deep	☐	TOC - shallow	☐	TOC	☐
Dissolved metals - shallow	☐	Total nutrients - shallow	☐	Total nutrients	☐
DOC - deep	☐	DOC - shallow	☐	DOC	☐
DOC - shallow	☐	Dissolved anions - shallow	☐	Dissolved anions	☐
TOC - shallow	☐	SUVA	☐	SUVA	☒
Total nutrients - deep	☐				
Total nutrients - shallow	☐				
Dissolved anions – deep	☐				
Dissolved anions – shallow	☐				
SUVA	☐				

Water Chemistry Lab	NOTES
USEPA ATTN: Venkatapathy 26 West Martin Luther King Dr. West Cincinnati, OH 45268 Phone: 513-569-7077 Email: Venkatapathy.raghuraman@epa.gov	Please note any problems from field collection and/or sample receiving (e.g. no deep samples collected because Van Dorn broke or samples were warm upon receiving):

RECEIVED FROM FIELD

Name:		Signature:		Date:	

RECEIVED FOR ANALYSIS

Name:		Signature:		Date:	

F10. SuRGE CHLOROPHYLL, PHYCOCYANIN, AND MICROCYSTIN TRACKING SHEET

SuRGE ALGAL INDICATOR SAMPLE TRACKING SHEET

SHIPPING INFORMATION

Sender: ______________________ Sender Phone: ______________________

Shipped by: ○ FedEx ○ UPS ○ Other: ______________________

Tracking Number: ______________________ Date Sent: ______________________

LAKE INFORMATION

Lake siteID: ______________________ Crew: ______________________

Date Collected: ______________________

SAMPLE INFORMATION

ROUTINE		DUPLICATES		BLANKS	
Sample type	Collected?	Sample type	Collected?	Sample type	Collected?
chlorophyll	☐	chlorophyll	☐	chlorophyll	☐
phycocyanin	☐	phycocyanin	☐	phycocyanin	☐
microcystin	☐	microcystin	☐	microcystin	☐

Water Chemistry Lab	NOTES
USEPA ATTN: Shivers 27 Tarzwell Drive Narragansett, RI 02882 Phone: 401-782-9629 Email: shivers.stephen@epa.gov Alternate: hollister.jeff@epa.gov	Please note any problems from field collection and/or sample receiving (e.g. microcystin bottle broke or samples were received warm):

RECEIVED FROM FIELD

Name:		Signature:		Date:	

RECEIVED FOR ANALYSIS

Name:		Signature:		Date:	

F11. SuRGE ALGAL TAXONOMY AND PHYSIOLOGY TRACKING SHEET

SuRGE ALGAL INDICATOR SAMPLE TRACKING SHEET

SHIPPING INFORMATION

Sender: ____________________ Sender Phone: ____________________

Shipped by: ○ FedEx ○ UPS ○ Other: ____________________

Tracking Number: ____________________ Date Sent: ____________________

LAKE INFORMATION

Lake siteID: ____________________ Crew: ____________________

Date Collected: ____________________

SAMPLE INFORMATION

ROUTINE	
Sample type	Collected?
Taxonomy (1 sample)	☐
Physiology (2 samples)	☐

Water Chemistry Lab	NOTES
USEPA ATTN: Tatters 1 Sabine Island Drive Gulf Breeze, FL 32561 Phone: 910-393-7078 Email: tatters.avery@epa.gov Alternate: aukamp.jessica@epa.gov	Please note any problems from field collection and/or sample receiving (e.g. sample leaked, inadvertently frozen, etc):

RECEIVED FROM FIELD

Name:		Signature:		Date:	

RECEIVED FOR ANALYSIS

Name:		Signature:		Date:	

F12. SuRGE GAS SAMPLE TRACKING SHEET

SHIPPING INFORMATION	
Sender: ______________________	Sender Phone: ______________
Shipped by: ○ FedEx ○ UPS ○ Other: ______________________	
Tracking Number: ______________________	Date Sent: ______________
Ship to : USEPA, ATTN: Daniels, 26 Martin Luther King West (MS 585), Cincinnati, OH 45268 e-mail: Daniels.Kit@epa.gov, phone: 513-376-1333	

GAS VIAL INFORMATION					
EXETAINER CODE	SAMPLE TYPE (AIR, DG, TRAP)	EXETAINER CODE	SAMPLE TYPE (AIR, DG, TRAP)	EXETAINER CODE	SAMPLE TYPE (AIR, DG, TRAP)

RECEIVED					
Name:		Signature:		Date:	

F13. LOS GATOS GREENHOUSE GAS ANALYZER QA/QC SHEET

Instrument model (UGGA or MGGA): ______________________

Instrument serial number: ______________________

Calibration standard lot # ______________________

					Calibration check			
Date	Lake siteID	Cell Pressure (Torr)	Laser A τ (ringdown time)	Laser B τ (ringdown time)	Cal check start time	Cal check end time	CH_4 after 1 minute (1.6 – 2.4)	CO_2 after 1 minute (320 – 480)

F14. PICARRO GREENHOUSE GAS ANALYZER QA/QC SHEET

		Calibration check								
		1-minute mean					1-minute precision			
Date	Lake siteID	Cal check start time	Cal check end time	CH_4 (ppm)	CO_2 (ppm)	N_2O (ppb)	CH_4 (ppm)	CO_2 (ppm)	N_2O (ppb)	Measurement interval

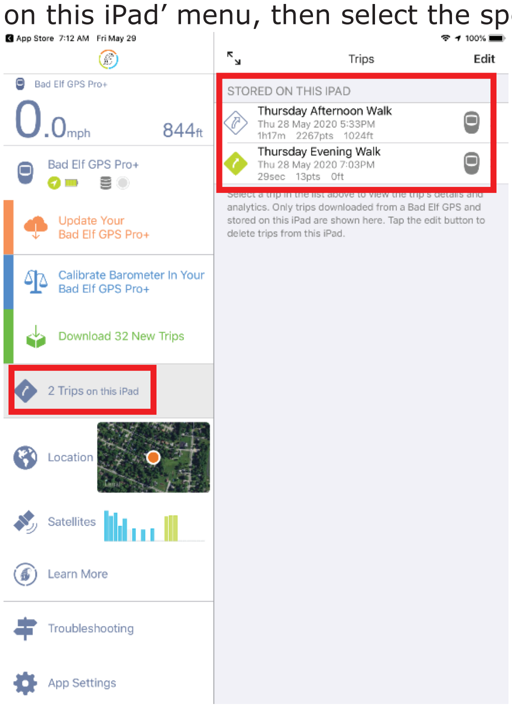
F15. Bad Elf GPS USER GUIDE

Activating data recording

Your position on the lake should be recorded continuously during floating chamber deployments using the provided Bad Elf GPS unit. Press and hold the GPS button (bottom left) for 3 seconds to turn on data logging. Once data logging is turned on, the LCD display shows a blinking icon along the bottom of the display. The Bad Elf GPS records your position every second while data logging is on.

Previewing recorded data

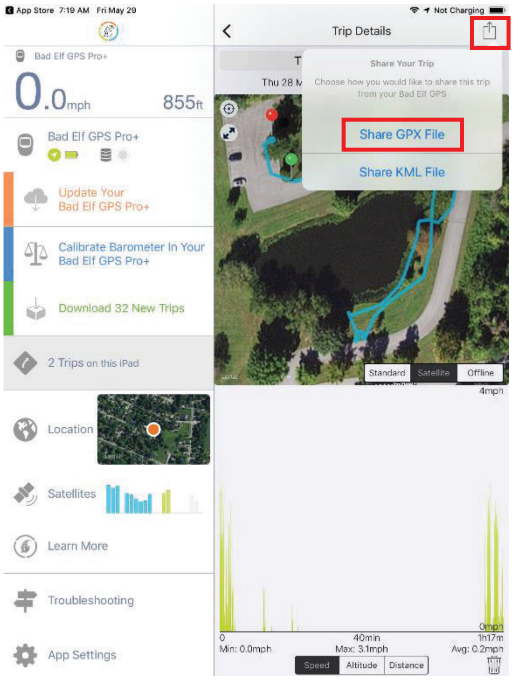
To stop recording data, press and hold the GPS button for 3 seconds and a prompt will appear. You can then choose either to record a point of interest (POI) and continue data logging or to turn data logging off. Data logs can be easily previewed using the Bad Elf app on your iOS device. With the GPS connected to your device, open the app, select the 'Trips on this iPad' menu, then select the specific track log you wish to preview.



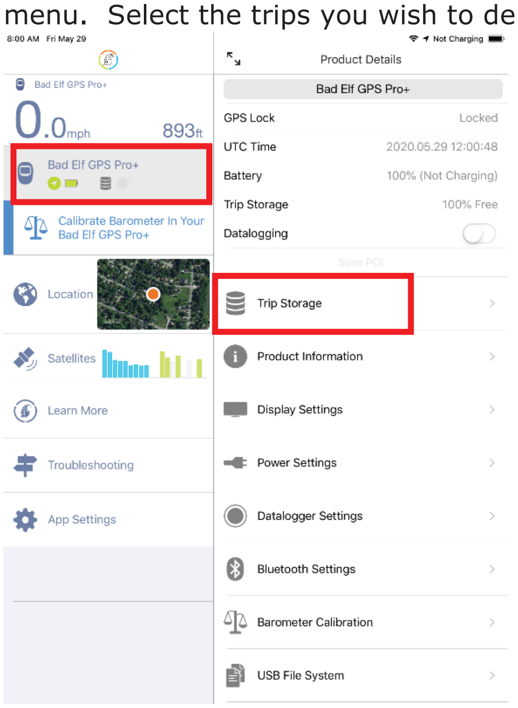
Downloading data

Once data logging is complete, you can use the Bad Elf Utility app to save, map, share, or delete the recorded trip. Alternatively, you can connect the GPS to your computer with a USB cable and access the log files directly through your file explorer. The Bad Elf Utility app is the preferred method.

To download via the app, select 'Share GPX File' from the overflow menu  while previewing a data log (see above). You have several options for 'sharing' the file, including e-mail and text message. It may be most convenient to simply e-mail the file to yourself. Each iPad has been preconfigured with a gmail account (see F16. GeoPlatform and Explorer Workflow) which will be used as the default e-mail.



Downloaded files can be removed from the GPS by selecting 'Trip Storage' from the GPS menu. Select the trips you wish to delete from the 'Manage Trips' menu and hit delete.



To download to computer, make sure the GPS is powered off, then connect to computer via USB cable. Select the 'USB drive' option when the GPS turns on. This will cause the GPS to generate .gpx data files containing the track logs. These can be downloaded from the 'logs' folder via Windows Explorer. Data file names will start with the date of collection (e.g. '2020-05-28'), followed by a 10-digit alpha numeric code.

[redacted]

(b) (6)

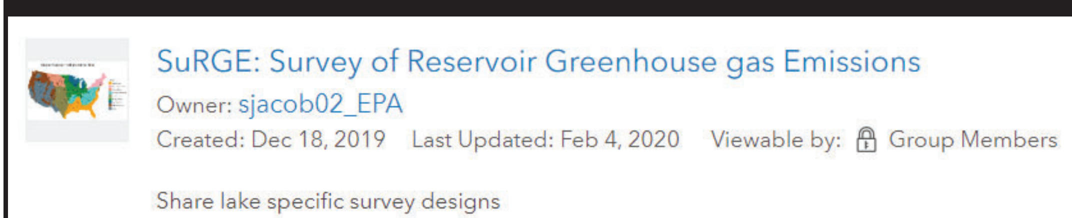
[redacted]

GeoPlatform

[redacted]

link

[redacted]

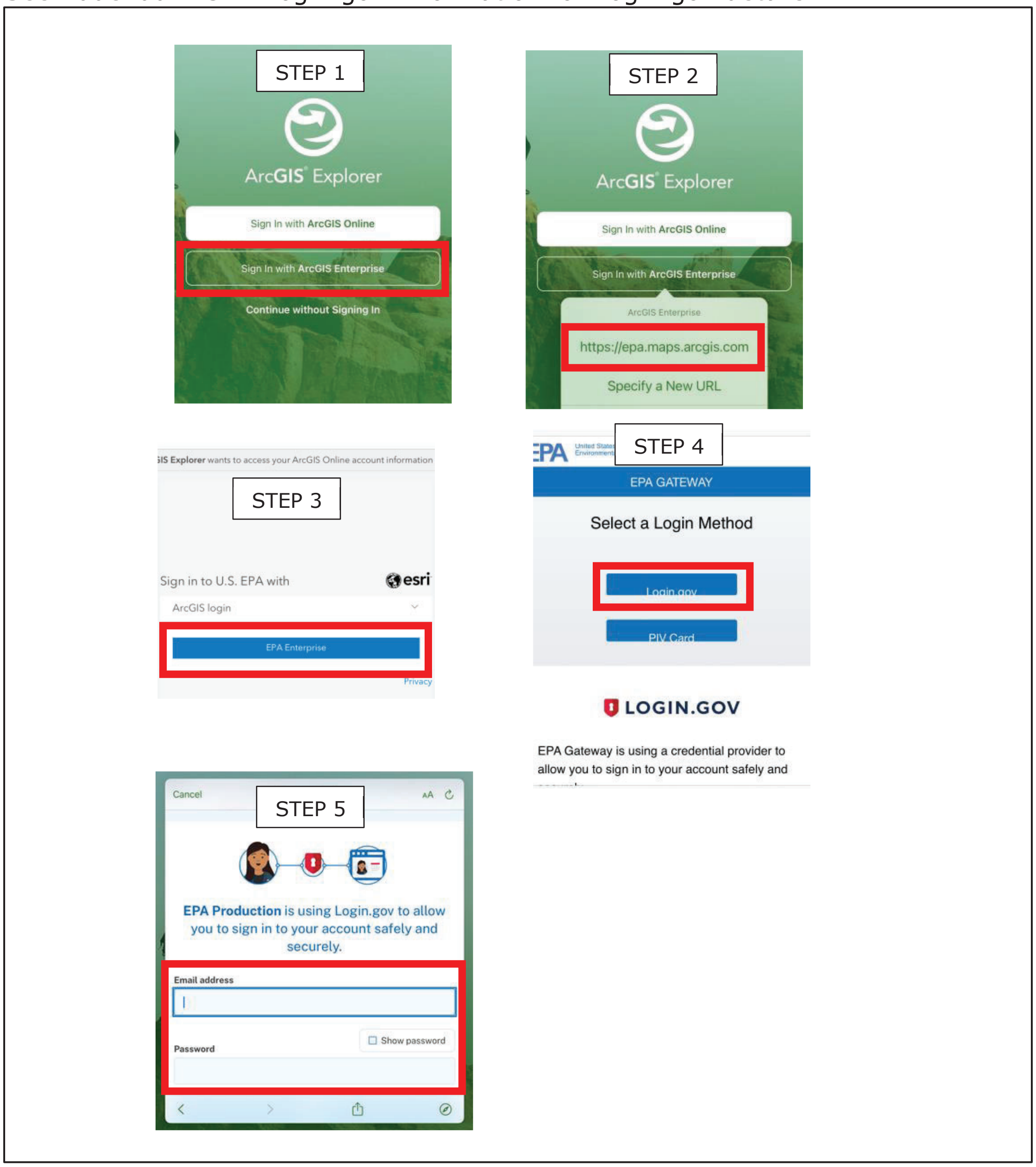


Explorer

Logging into 'Explorer'

[redacted]

3. If you have an @epa.gov e-mail address, you select 'Sign in with ArcGIS Enterprise', specify or choose 'epa.maps.arcgis.com', then select 'EPA Enterprise'. In step 4, you must choose 'Login.gov' (unless you can interface a card reader to the iPad). Follow the prompts, then provide your @epa.gov email address and Login.gov password. Note that the Login.gov password might not be the same as your LAN password. See Addendum G2. Login.gov Information for Login.gov details.

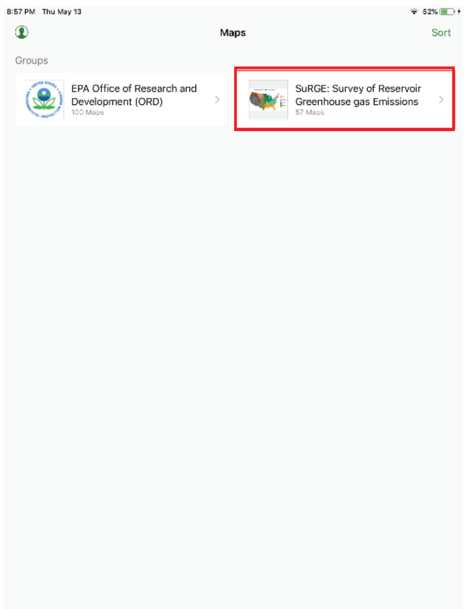


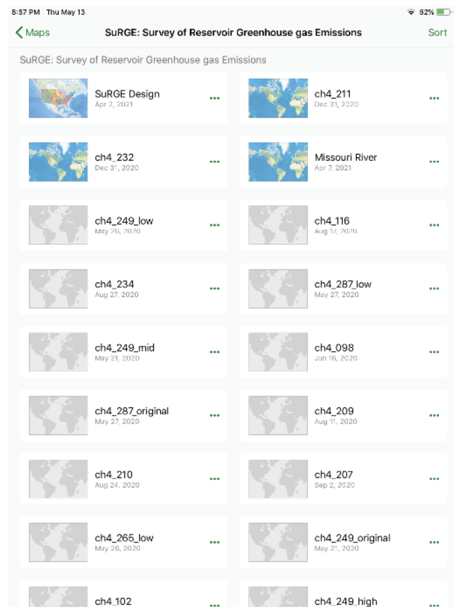
4. Outside collaborators must sign in using the 'ArcGIS Online' option and the credentials provided by EPA Geoservices support team.

Procedure for downloading maps

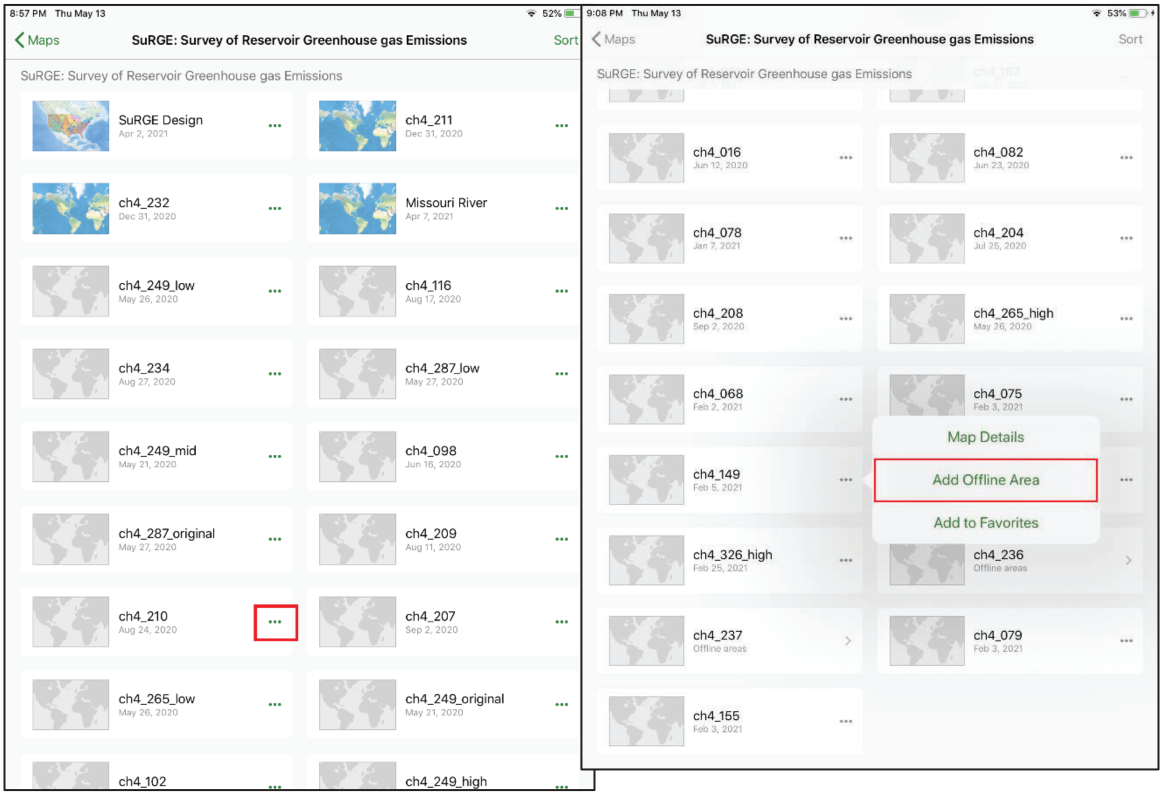
See Logging into 'Explorer' above for log in details.

1. A Wi-Fi or cellular data connection is required to download maps. If using cellular data, please click the profile button in the top left of Explorer, and make sure "Only Download on Wi-Fi" is toggled to the left. This way, cellular data will be used when Wi-Fi is not available.
2. By default, the app will show all maps that you have access to via the GeoPlatform. To narrow the list, scroll to the bottom and select the SuRGE group. You will now see a menu of all SuRGE lake designs identified by unique SuRGE lake siteID.

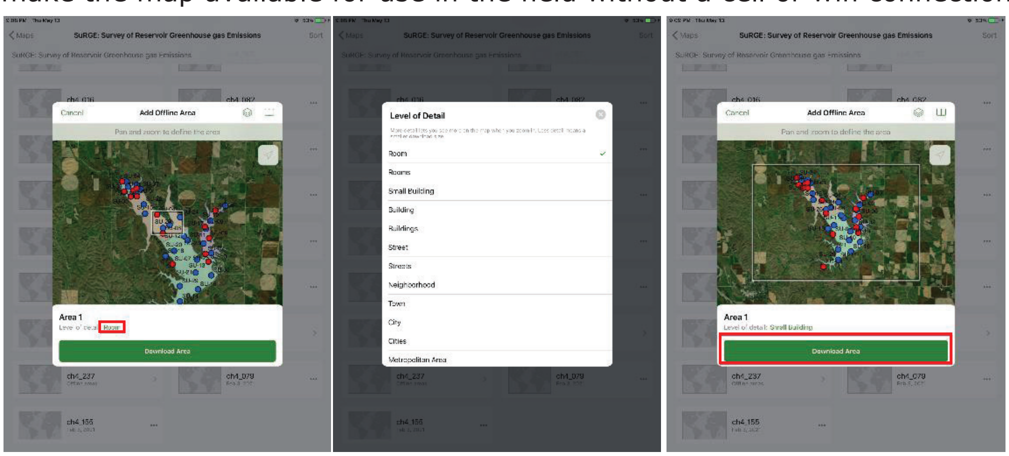




3. To download a map for use in the field, tap on the three blue dots adjacent to each lake name and select 'Add Offline Area' in the new menu.

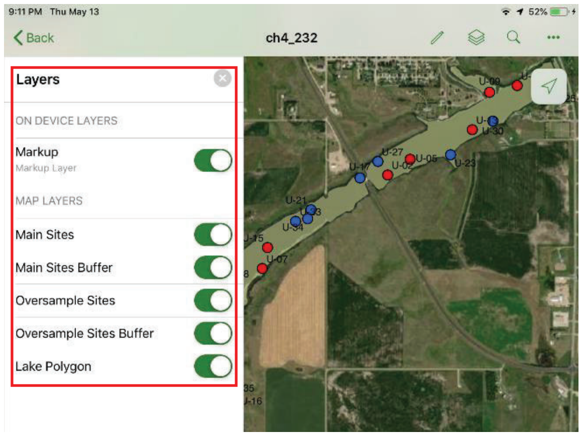


4. The map extent to be downloaded is determined by the 'Level of Detail' option. Choices range from 'Room' for a small extent, up to 'World'. Choose the smallest 'Level of Detail' that encompasses the entire lake. Finally, pan the map such that the entire lake fits within the highlighted area and tap 'Download Area'. This will make the map available for use in the field without a cell or wifi connection.



5. If you wish to preview the design prior arriving at the lake and the iPad is not connected to an integrated or Bluetooth GPS, simply tap the lake name.

6. If you wish to preview the design prior arriving at the lake and the iPad is connected to an integrated GPS or Bluetooth GPS, the map view will default to your current location.
    a. To change the map view to show the lake,..
        i. Tap the layers icon in the top right corner 
        ii. Tap any of the layers in the new Layers menu.



        iii. This will change the map extent to that of the lake design.

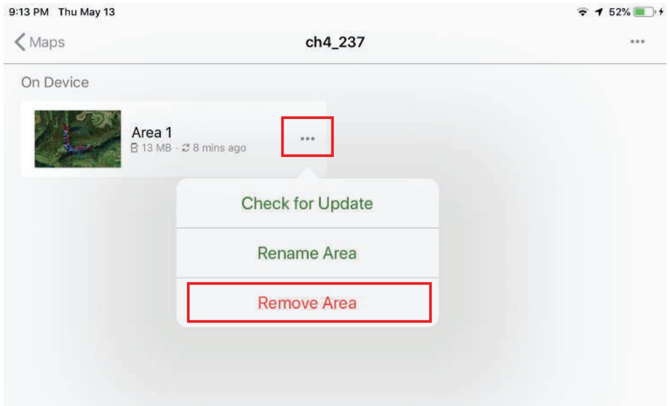
Procedure for locating sites in the field

1. Connect iPad to provided Bad Elf Bluetooth GPS or equivalent.
2. Open Explorer and tap on the downloaded map.
3. The app will show your location as a blue dot surrounded by a hollow circle with a blue outline. The blue outline indicates the GPS accuracy.
4. Target sample sites are shown in red and oversample sites in blue. If you zoom in close enough, you will notice that each sample site is nested within a grey circle. The grey circle indicates the required accuracy for sampling. As long as your location is within the grey circle, you may conduct the sampling.
5. Tap the layers icon  in the top right to open the layers menu.
    a. You may turn on and off the layers at your discretion.
6. Tap the three blue dots at the top right to view more options, including an option to turn on the legend.

Procedure for removing maps from device

1. When you are finished with a downloaded map, it is good practice to remove it from the device to conserve storage space.
2. Tap the map you want to remove.

3. Select 'Remove Area' from the overflow menu •••



G. Addendum

G1. COVID Related Delays in Sample Analysis During 2020

COVID related social distancing policies caused most EPA analytical facilities to close during the 2020 SuRGE sampling season. As a result, sample holding times will be exceeded for the following analytes: dissolved metals, dissolved nutrients, total nutrients, TOC, and gases. We do not anticipate holding time issues with the three algal indicators.

Any sample that exceeds the holding time will be flagged. Below is a discussion of the potential impacts of holding time violations.

Dissolved Metals

Dissolved metals are preserved with 3 drops of 2% nitric acid and have a holding time of 60 days at room temperature. The analytical method further acidifies the samples to ensure all metals are fully dissolved and quantifies the total mass of up to 24 species. The analytical method isn't sensitive to the chemical form of the metal (e.g. $Fe(OH)_3$ vs Fe^{++}), therefore any biotic or abiotic reactions causing metals to change form during sample storage will not affect the analysis. The dissolved metals analysis is extremely robust to sample storage issues and we do not anticipate data quality issues associated holding time violations.

Dissolved and Total Nutrients

Total and dissolved nutrient samples (phosphorus and nitrogen) have a 24 hour holding time at 5°C and 28 days at -20°C. Dissolved nutrients are defined as those that pass through a 0.45μm filter, whereas Total nutrients include both particulate and dissolved forms. The analytical method for Total nutrients analysis uses digestion procedures to convert all nutrients (dissolved and particulate) to dissolved inorganic species, therefore any conversion between nutrient species during sample storage will have very little effect on the Total nutrient analysis. A separate storage issue that could affect the dissolved forms is adsorption to the inner walls of the sample container. While it isn't clear if this is a time dependent process, a brief literature review indicated that this effect can reduce the true dissolved concentration by up to 10%. This effect is quite small when compared to the anticipated range of dissolved and total nutrient concentrations expected across the 108 SuRGE sites (0.01 – 10 mg TN/L, 0.01 – 0.8 mg TP/L). This storage effect will not greatly affect our ability to use nutrient concentrations to separate sites by trophic status and nutrient availability.

TOC

Total organic carbon samples are preserved with acid (pH<2) and stored at 5°C for up to 28 days. The analytical procedure further suppresses the pH to drive off any inorganic carbon. All remaining organic is oxidized to carbon dioxide and quantified via non-dispersive infrared detection. TOC analysis is insensitive to conversions between different organic carbon forms during storage, but oxidation of organic carbon to inorganic forms during storage is a concern. The low pH should arrest biological activity during storage, but abiotic reactions cannot be entirely ruled out.

To minimize TOC holding time violations, all 2020 SuRGE TOC samples will be analyzed by an outside contract laboratory (MASI Laboratory), rather than being stored until EPA facilities reopen. This decision was made part way through the 2020 sampling season. MASI has provided TOC vials pre-acidified with phosphoric acid and requires all samples to be collected in duplicate. MASI will also analyze the sulfuric acid preserved samples collected earlier in the study.

All 2020 SuRGE TOC samples will be shipped to the Cincinnati EPA location, then repackaged and driven to MASI's Cincinnati laboratory. MASI analyzes TOC using UV persulfate oxidation and non-dispersive infrared detection (SOP WET 18). The EPA method also uses non-dispersive infrared detection, but oxidizes the samples using catalytic combustion at 680°C. Both techniques are widely used and we don't anticipate any problems with comparability between laboratories. MASI also follows strict QA/QC requirements, similar to that of USEPA.

Gases

Gas samples are collected in over-pressurized glass vials capped with PTFE/silicone septa, chlorobutyl septa, and screw top lids. The chlorobutyl septa forms a seal on top of the glass vial while the PTFE/silicone septa ensure that needle punctures do not compromise the seal. The storage time for gas samples is 4 months at room temperature.

During sample storage, gases will diffuse into and out of the storage vials via diffusion, causing the sample contents to slowly equilibrate with those of the surrounding atmosphere. Gas samples collected from passive gas traps will contain a methane (CH_4) partial pressure (~60%) that greatly exceeds that of the atmosphere and a dinitrogen (N_2) partial pressure much lower than that of the atmosphere (~30%). Thus storage will increase N2 and decrease CH4 in the sample. Rates of diffusion through glass vials are exceedingly slow, however. For example, Hamilton and Ostrom (2007) showed no discernible change in sample N2 content in up to 60 days of sample storage using only a chlorobutyl septa. We expect even slower rates of gas exchange with the addition of the PTFE/silicone septa.

We expect any sample storage effect to be minimal compared to the range of concentrations expected in SuRGE gas samples (5-90% CH_4 in passive trap samples). We will conduct a sample storage test to better quantify this effect.

G2. Login.gov Information

The document embedded below in the Word version of this QAPP details how to access the GeoPlatform Online through the new EPA gateway login page on a number of different types of devices (computers inside or outside the EPA, iPhones, etc.). **For the pdf version of this QAPP, the document is appended, see the next page.**

G2_Login_Gov_forGeoPlatformUsers.pd

Login.gov information

GeoPlatform users, as you may (or may not) be aware, public access to EPA systems is migrating away from the current WAA (Web Application Access) Username & Password solution to the EPA Gateway, which is powered by Login.gov. This migration was completed on January 13, 2022 and impacts how users log into the EPA GeoPlatform Online (GPO) for multiple scenarios outlined below.

GPO login from computers inside the EPA network

There will be no change to the GPO login process for Windows users within the EPA network as a result of the Login.gov migration.

GPO login from computers outside the EPA network

As of January 13th, 2022, the new EPA gateway login page was implemented and login via Login.gov is the only option. If you have more than one email address associated with your login.gov account, please make sure to use your EPA email address as your username when logging in.

GPO Login through EPA-issued iPhone

Currently, users can no longer log into the GPO while on the EPA VPN from an EPA-issued iPhone. A temporary workaround is to disconnect from the VPN prior to logging into the GPO. Once the Login.gov migration is complete, users connecting to the GPO through an EPA-issued iPhone will be required to login with a Login.gov account.

For continued access to the GPO, EPA users are strongly encouraged to register for a Login.gov account by following the instructions in the attached email (at the end of this document).

Additional Frequently Asked Questions can be found in the next section.

Questions about login.gov should be directed to EISD@epa.gov. Additional questions about the GPO login can be sent to geoservices@epa.gov.

Frequently Asked Questions:

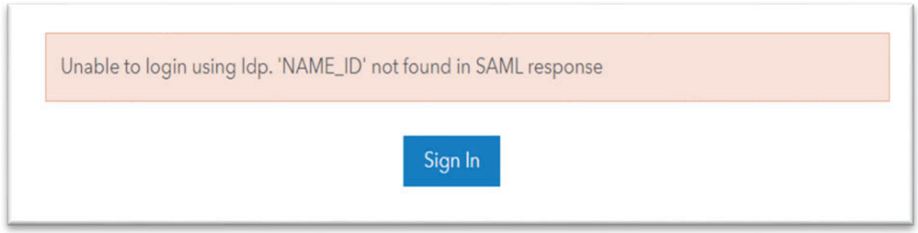
When did this change happen?

- The change to Login.gov for EPA Enterprise users was January 13, 2022.

How do I link my Login.gov account to my EPA Account?

- This process is being managed by the Enterprise Identity and Access Management Team and EISD. Follow below step on WAA's FAQ page:
  https://wamssoprd.epa.gov/customloginpage/pages/help/faq.jsp#logingov-account
  Or, this Help link:
  https://wamssoprd.epa.gov/customloginpage/pages/help/helpfiles/How_to_Register_a_New_WAA_EPA_Community_Account_with_Login_gov_Credentials.pdf

I received an error message like this when I tried using login.gov, what went wrong?

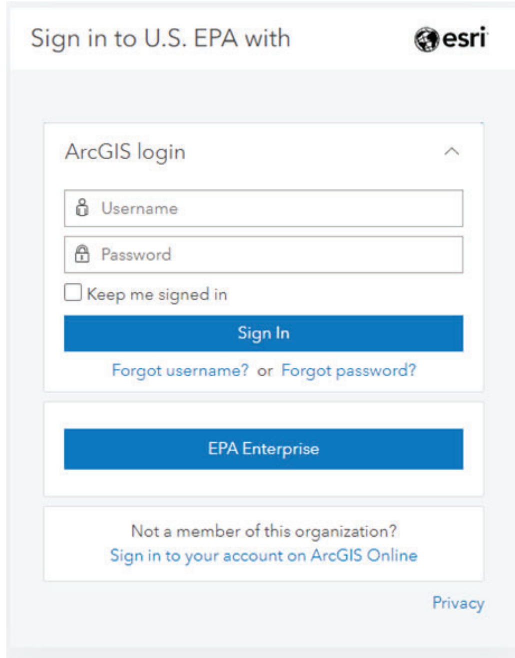


- This error occurs when someone uses a non-EPA email address as their username at login.gov. If you have an EPA.gov email address, please sign out of login.gov and sign in again using your EPA email address as your username.
- If you do not have an EPA.gov email address, you should not be using "EPA Enterprise" and login.gov to authenticate to the EPA GeoPlatform. Instead, you should be using the ArcGIS login option.

I am still getting errors trying to log in because my accounts aren't linked - what now?

- If your account is not linked after following the steps in the help pages linked above, try the following steps:
    - Disconnect from the EPA intranet (Pulse VPN) on your EPA-issued computer (GFE).
    - Log into https://waa.epa.gov using the login.gov option. Use your EPA email as the ID and your Login.gov password.
    - When prompted for a secondary authentication:
        - If prompted for your EPA PIV, follow the steps to sign in with your PIV card.
        - If no EPA PIV prompt is provided, go to the other secondary authentication page and select EPA PIV (Federal employee ID). Then follow the steps to sign in.
    - Next time you log in you should be able to have your accounts linked. You can test your login from a second type of browser window – for example, select Edge if you first used Chrome to set up the PIV card.
    - If you are still having issues, email EISD@epa.gov.

Does this change to Login.gov impact, not just EPA users, but all the GPO accounts as we are transitioning to Login.gov? For example, START and State partners – will they need to make login.gov accounts as well?



- The first email sent about the change was intended to apply only to those GeoPlatform accounts who choose the "EPA Enterprise" login option and use their EPA LAN credentials for authentication. **All GeoPlatform accounts who choose the "ArcGIS Login" option are unaffected by the login.gov, and their login workflow is unchanged.** Currently, we do not recommend that external GeoPlatform users switch to login.gov authentication.

- Note: We understand that Esri is independently working on login.gov integration for all of ArcGIS Online, and there may come a point in the future where these ArcGIS logins will also need to transition to login.gov, but fortunately, that's not a concern yet.

I've heard there are different "tiers" of Login.gov users requiring different levels of authentication (such as just an email vs. needing a scanned driver's license). Are all GPO users being treated as the same tier? What tier is the EPA GPO and does that affect the registration instructions I should be using?

- The GeoPlatform owners submitted a DIRA (Digital Identity Risk Assessments) form stating the EPA GPO IAL level was 1 – the lowest level. This level does not require identity-proofing and all users will fall into the same tier. While we expect no changes, this DIRA has not been completely approved and we will notify all users if this changes.

Our Program/Office has created separate project-related accounts on GPO to use with various applications. Is there a strategy for how to handle those types of existing accounts with the new login.gov?

- These accounts are very common and in widespread use. They are both practical, and in some cases, critical to workflows. As with other external accounts, these project-related accounts use the ArcGIS logins and will not need login.gov at this time.

- When Esri introduces their login.gov integration, we will place an extremely high emphasis on maintaining system accounts in some capacity.

Contractors off the EPA VPN still are unable to access the Dashboard for requesting public approval of content. Are there any plans to make the Dashboard more accessible?

- We're just a few weeks out from the Dashboard being accessible at a public URL.

EMAIL Sent about Login.gov with instructions:

On November 1, 2021, access to EPA resources will change. All external users will be required to login via the EPA Gateway powered by Login.gov. ***As an external user, you must create a Login.gov account by December 31, 2021 to maintain your access to EPA resources.***

Background
In accordance to Executive Order 13681, Login.gov is a government-wide solution that offers the public secure, private, streamlined access. Rather than requiring individuals to have a separate login for each federal agency's electronic system, the General Services Administration's (GSA's) Login.gov allows individuals to access information or request services from any of its participating federal agencies. Registered users can simply and securely sign into multiple government agencies with a single Login.gov user ID and password.

Login.gov will serve as the identity provider and therefore, will collect the information necessary to complete identify proofing. Login.gov has the capability to meet the identity assurance levels (IALs) described in NIST-800-63 and the EPA Gateway will leverage these identities to facilitate secure access. The information requested by the system and asserted back to the agency is only what is necessary to establish access at the appropriate assurance levels.

What does this mean for me?

The logon page previously used for accessing EPA WAA Community Applications will be changing.

Your EPA-issued ID and password will expire on December 31, 2021. To continue accessing EPA resources, you must follow a three-step process to create a Login.gov account and link it to your EPA application account.
Important: You must use the same email address that is associated with your EPA-issued account when creating your new Login.gov account.

Step 1: Using your EPA-issued username/password, login to the EPA Web Application Management Dashboard and click User Profile to verify the email address associated with your account.

- *How to Lookup EPA WAA Community Email Address*

Step 2: Create a Login.gov account (You must use the same email address found in Step 1.)
NOTE: If you already have a Login.gov account you can change the email address in your EPA User Profile to match your current Login.gov email address in Step 1.

Step 3: Follow the instructions here to link your Login.gov credentials to your EPA application account.

Once you have completed this three-step process, you may go to the desired application and use your new Login.gov credentials to more securely login to EPA resources.

Does the EPA Gateway allow me to use my EPA-issued Username and Password to access applications?

Access to EPA applications using the WAA Username and Password method will continue to be available for a limited time via the EPA Gateway website. This authentication option will be retired on December 31st, 2021.

- *How to Log into EPA WAA Community Access with WAA Username and Password*
- *How to Log into EPA WAA Community Applications with WAA Username and Password*

How do I get help?

For issues with Login.gov accounts (e.g., password resets, inability to log in with Login.gov credentials) go to Login.gov's support. If you no longer have access to an agency application, please contact the application sponsor. All other questions may be sent to the EPA Help Desk (EISD@epa.gov).

Enterprise Identity and Access Management Team - Environmental Protection Agency